THE JOURNAL
OF THE
AMERICAN LEATHER CHEMISTS ASSOCIATION

VOL. LI AUGUST 1956 No. 8

CONTENTS

Reaction of Nitrous Acid with Collagen *By* J. M. CASSEL - 406

Bonding Film Adhesion Test for Leather Finishes
By C. A. BURKART, N. D. GALLAGHER, and M. B. NEHER 418

Report of the Leather Finishes Committee - - - 429

ALCA News - - - - - - - - - - - - - 431

Abstracts - - - - - - - - - - - - - - 443

Patent - - - - - - - - - - - - - - 456

New Publications - - - - - - - - - - 456

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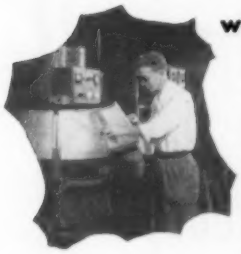

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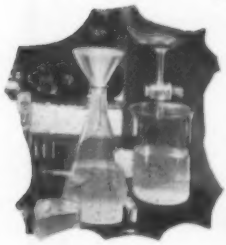

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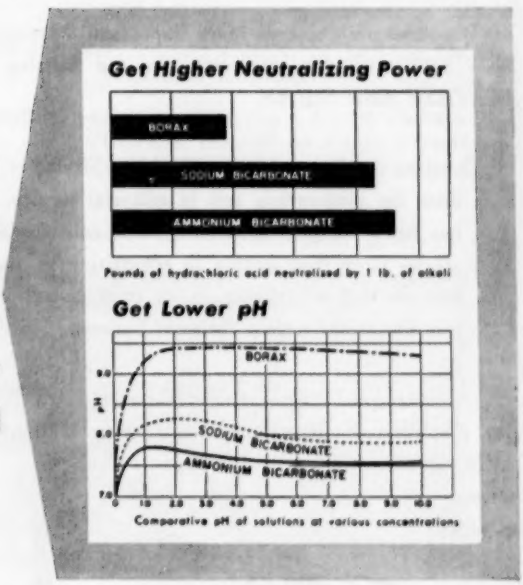

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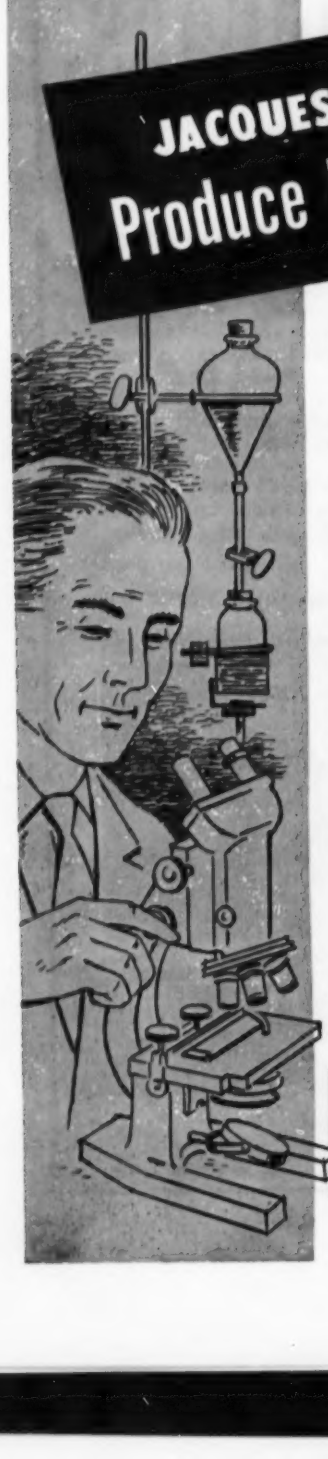

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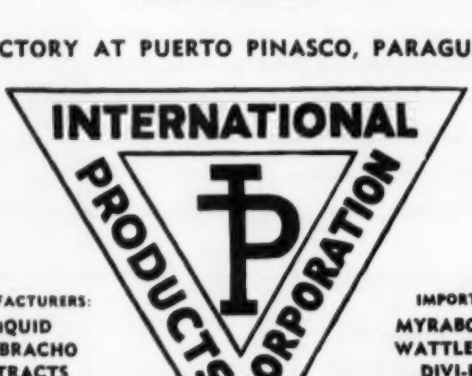

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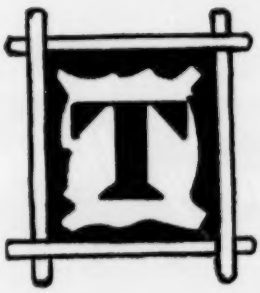

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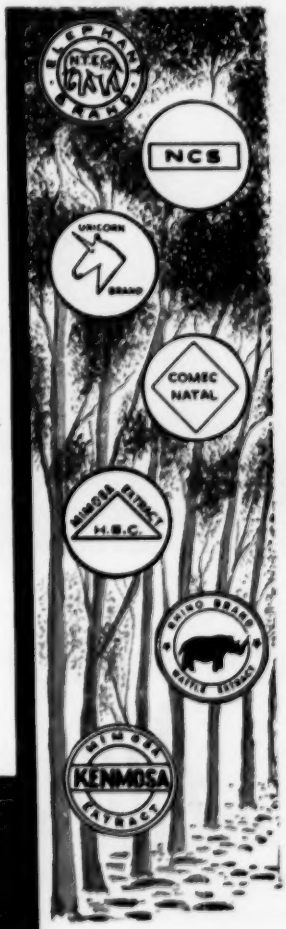

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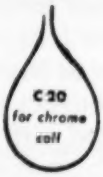

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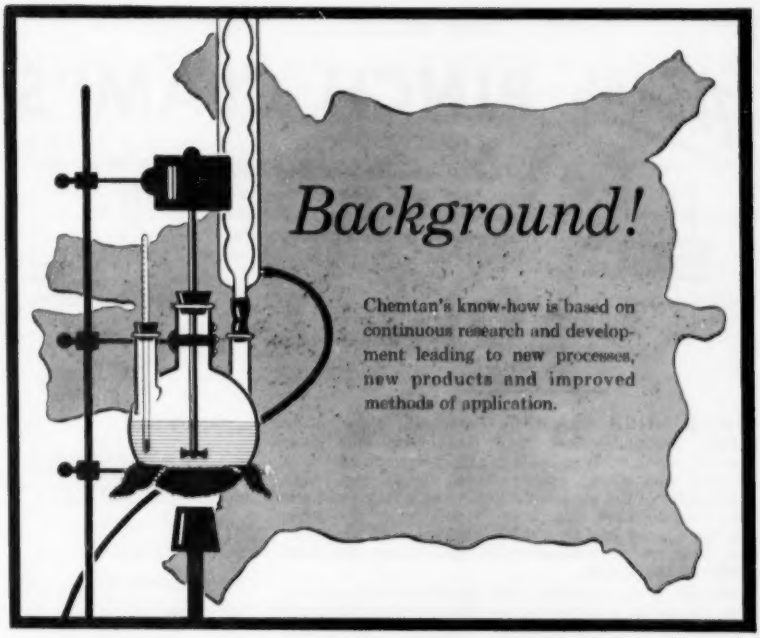
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Fig. 1. Showing appearance of Sizes 12 and 18. Joint held securely by spring.

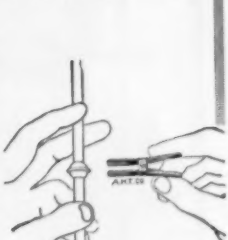

Fig. 2. Showing method of attaching to joint. Parts held securely by spring upon release of finger pressure.

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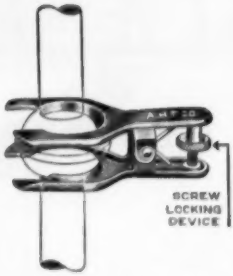

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Reaction of Nitrous Acid With Collagen*

JAMES M. CASSEL

National Bureau of Standards,
Washington 25, D. C.

ABSTRACT

The reaction between nitrous acid and collagen at 16° C. was studied by determining the amino acid composition of derivatives prepared by varying the time of the reaction from five minutes to six days. One sample, after reacting with nitrous acid, was decolorized by treatment with permanganate and bisulfite. After hydrolysis the amino acid analyses of all derivatives were obtained by two-dimensional paper chromatography. Aspartic acid, glutamic acid, glycine, proline, hydroxyproline, leucine, isoleucine, valine, alanine, phenylalanine, and histidine were not attacked by nitrous acid. The ε-amino, guanidino, and phenolic groups of lysine, arginine, and tyrosine, respectively, were attacked, but not to the same degree. The lysine content decreased progressively to zero. In samples deaminated for periods longer than 12 hours the arginine content was reduced. The tyrosine content was decreased progressively by deamination, and both methionine and tyrosine were attacked by the decolorizing procedure.

INTRODUCTION

The chemical and biological activity, as well as the structure of protein, are to a great extent dependent on the amino acid polar residues projecting as side groups from the main chain (1, 2). One such group is the lysine residue containing a free amino group at its extremity. A favorite method of studying these amino groups to evaluate their role in protein structure and various protein reactions has been to remove them by reaction with nitrous acid. Excellent reviews of the literature on the reaction of nitrous acid with proteins have been given by Dunn and Lewis (3), and Olcott and Fraenkel-Conrat (4).

Numerous investigators have treated collagen, gelatin, hide powder or hide sections with nitrous acid. Hitchcock deaminated gelatin and showed by acid-binding determinations that only a portion of the basic groups of the protein were attacked by nitrous acid (5). Atkin (6) studied the effect of deamination on the complete titration curve of hide powder. Cassel and Kanagy (7) measured the electrophoretic mobilities of deaminated hide powder particles.

Collagens treated with nitrous acid have been used frequently in attempts to study the mechanism of tanning. Meunier and Schweikert (8,) Highberger and Retzch (9), and Bowes and Pleass (10) all compared the combination of formaldehyde with deaminated and with untreated hide powder. Thomas and Foster (11), Page and Holland (12), Lollar (13), and Bowes and Kenten

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(14) used deaminated hide powders in studying the mechanism of vegetable tanning. Gustavson (15) used them in a study of cationic chrome fixation.

The reaction of nitrous acid with the amino groups of soluble proteins is rapid, and side reactions are not likely to occur. However, the reaction with soluble protein is not entirely specific for the ε-amino group as partial destruction of cystine, tyrosine, and arginine has been reported (16, 3). In contrast to soluble proteins, collagen is a fibrous, practically insoluble material. This difference in physical state requires that time be allowed for the nitrous acid to diffuse into the interior of the fiber, and it is possible that groups other than lysine residues may be affected. Stubbings and Theis (17) reported that arginine residues of collagen were not attacked in one day of deamination but that in one week about 50% destruction had occurred. Histidine was unattacked. Bowes and Kenten (14) observed a 15-20% loss of arginine after a nitrous acid treatment for two days. Cassel, McKenna, and Glime (18) also reported loss in arginine on prolonged nitrous acid treatment.

None of the investigators referred to determined the effect, if any, of nitrous acid on amino acid residues other than those of the basic and acidic amino acids. Because the deamination reaction is likely to be used as much in the future as in the past, a critical study and analysis of the protein residue derived from reaction with nitrous acid is of value. For this reason, a series of derivatives were prepared by treating the collagen with nitrous acid for varying periods of time. The degree of deamination as well as the extent of other reactions which may have occurred was determined by analysis of the products.

MATERIALS AND METHODS

The parent collagen used in the deamination reaction was a highly purified material prepared from a steer hide obtained one hour after the death of the animal. The grain and flesh layers of the hide were split off, after which the corium was ground to a powder and freed of extraneous fats, salt, and proteins by a series of aqueous and organic extractions (19). The final product was ground in a Wiley mill to pass a 4-mm. sieve.

Collagen was treated with nitrous acid according to the method of Thomas and Foster (11) as modified by Bowes and Kenten (14). For samples allowed to react for an hour or less the following procedure was used: Collagen (150 g.) was soaked overnight in a liter of distilled water at 16° C. Sodium nitrite (150 g.) was dissolved by stirring it with the collagen-water mixture, and 150 ml. of cold glacial acetic acid was added. The reaction vessel was kept at 16° C. to minimize side reactions (20). The mixture was stirred continuously throughout the first hour. At selected intervals a portion of the contents was removed and transferred to a sintered glass funnel where the washing procedure commenced immediately.

When samples were treated longer than one hour, CO_2 was bubbled through the reaction vessel to minimize oxidation of nitrous acid to nitric acid and to provide agitation (14). For each day the reaction was allowed to proceed, a fresh portion of nitrite and acetic acid was added. The products, initially yellow upon removal from the reaction vessel, were washed thoroughly with tap water and distilled water, and afterward dehydrated with several extractions in alcohol and acetone. When dried in air, the samples exhibited a range of color from yellow through straw brown to a reddish brown.

It has been reported that deaminated collagen can be decolorized by treatment with acid permanganate. To study the effect of this decolorizing treatment on the amino acids of deaminated collagen a sample, deaminated for 23 hours, was decolorized by a procedure similar to that suggested by Stubbings and Theis (17). To the deaminated product was added a 0.005 *M* solution of potassium permanganate in 0.1 *N* sulfuric acid. After stirring well, the mixture was filtered and rinsed several times with distilled water. The black fibers were then stirred with a one % solution of sodium bisulfite in order to remove the manganese dioxide that had been precipitated. The fibers were again washed with water. This entire procedure was repeated a number of times until most of the brown deaminated material had been converted to a white product that resembled the native untreated collagen. The decolorized material was thoroughly washed in distilled water, dehydrated by extractions with alcohol and acetone, and dried in air at room temperature.

The amino acid analyses of the deaminated and decolorized samples were obtained by application of a two-dimensional paper chromatographic technique described in a pervious paper (21). Microaliquots of hydrochloric acid hydrolysates of the deaminated materials were applied to Whatman No. 1 paper. The chromatograms were irrigated in the first direction with a 20:5:1 mixture of methyl alcohol, water, and pyridine, and in the second direction with a 10:10:5:1 mixture of tertiary butyl alcohol, methylelthylketone, water, and diethylamine. Before spraying with ninhydrin the sheets were steamed (21) to remove adsorbed diethylamine. Color was developed at 25° C. and 37% relative humidity. The amino acid-ninhydrin spots were cut out and extracted with aqueous *n*-propanol. Optical densities of the resulting colored solutions were read with a spectrophotometer set at 570 μ for all amino acids except proline and hydroxyproline which were read at 350 μ (22). Quantitative analysis was made by reference to standard plots of optical density versus concentration for each of the amino acids. The data in the standard plot were obtained from chromatograms of a synthetic amino acid mixture, the composition of which paralleled that found in a collagen hydrolysate.

The tyrosine contents of the products were also determined according to the Bernhardt (23) modification of the Millon-Weiss color reaction.

The methionine content of the decolorized sample was checked by the McCarthy-Sullivan procedure (24) as modified by Hess and Sullivan (25).

RESULTS AND DISCUSSION

Table I shows the nitrogen contents of the deaminated samples. These values were determined by the A. L. C. A. official method for nitrogen and

TABLE I

NITROGEN CONTENTS OF DEAMINATED COLLAGEN DERIVATIVES

Period of Deamination	Nitrogen Value % (dry basis)
0	18.1
5 min.	18.1
15 min.	18.1
30 min.	17.9
60 min.	17.9
4 hr.	17.7
12 hr.	17.6
23 hr.	17.7
32 hr.	17.7
50 hr.	17.9
6 days	17.3
23 hr. and decolorized	17.6

hide substance using mercuric oxide as a catalyst. It was found that a small continuous decrease in the nitrogen contents of the deaminated materials occurred through a period of six days with the exception of the sample treated for 50 hours. This contained slightly less nitrogen than the original material but the same amount as the sample deaminated 30 minutes. Since it is shown later that the lysine amino nitrogen is almost immediately attacked, the fact that the total nitrogen of the samples deaminated for 5 to 15 minutes was not reduced must indicate almost immediate combination with the protein by diazotization. For this reason it is difficult to assign any significance to the variation in nitrogen contents of the derivatives.

In Table II is listed a portion of the data obtained by chromatographic analysis of the collagen derivatives prepared by reaction with nitrous acid. A minimum of four chromatograms were run to obtain the values given in this table. The data indicate that neither nitrous acid treatment for as much as a week nor the decolorizing treatment caused any significant change in the percentages of the amino acids listed. The values for the two imino acids, proline and hydroxyproline, are observed to be somewhat increased

TABLE II

PARTIAL AMINO ACID ANALYSIS OF COLLAGEN DERIVATIVES

(Amino Acids not changed by Deamination)

Amino acid	Period of Deamination											
	0 min. %	5 min. %	15 min. %	30 min. %	60 hr. %	4 hr. %	12 hr. %	23 hr. %	32 hr. %	50 hr. %	6 days %	23 hr. + Decolorizing %
Aspartic acid	6.6	6.5	6.7	6.6	6.3	7.4	7.6	7.0	6.4	6.3	6.6	6.4
Glutamic acid	11.3	11.6	11.2	11.4	10.9	11.8	11.2	11.2	10.3	11.1	10.9	11.2
Glycine	26.1	26.1	25.4	27.0	25.3	28.0	30.4	27.8	25.6	24.9	24.6	25.2
Phenylalanine	2.9	2.6	2.9	2.9	2.8	——	2.3	2.7	2.9	3.0	2.7	2.5
Leucine + Isoleucine	5.6	5.6	5.4	5.8	5.5	5.6	4.5	——	4.5	5.9	5.6	5.7
Valine	3.0	2.8	2.8	3.1	3.0	2.8	2.8	3.4	3.1	3.2	3.0	2.8
Alanine	10.1	11.0	10.2	10.5	10.0	10.2	10.3	9.6	10.1	9.8	9.9	10.6
Hydroxyproline	12.4	12.5	12.6	12.9	12.6	13.1	12.3	15.0	——	13.9	14.1	13.0
Proline	14.8	14.8	14.5	14.8	14.1	14.6	——	——	14.0	16.2	15.5	18.0
Histidine	0.8							1.0		0.8	1.0	0.9

in the extensively deaminated samples. The proline value is obviously high in the sample that was decolorized. These increases are considered to be caused by increasing interference in the determination of these amino acids due to the formation of various degradation products that, on the chromatograms, had R_f values causing them to coincide with hydroxyproline and proline. Blue spots overlapping these two amino acids were sometimes apparent on the chromatograms of the extensively deaminated samples.

Previously, it had been shown (18) that exposure of collagen to nitrous acid for a week did not significantly alter the glutamic acid, aspartic acid, and histidine. The data of Table II confirm this finding. The advantage of the present method is that eight additional amino acids are shown to be unaffected by either the nitrous acid treatment or the decolorizing procedure.

It should be pointed out that amino acids altered in some manner by reaction with nitrous acid but reconverted by the conditions used subsequently for complete protein hydrolysis would be included in Table II. However, there is no evidence to indicate that such reactions occur.

In Table III are listed the amino acids whose residues in the protein peptide chains were in some manner altered by the treatment with nitrous acid or by the decolorizing procedure. The values listed in the table represent the average of a minimum of four chromatograms run on single hydrolysates. A discussion of the amino acids of Table III follows:

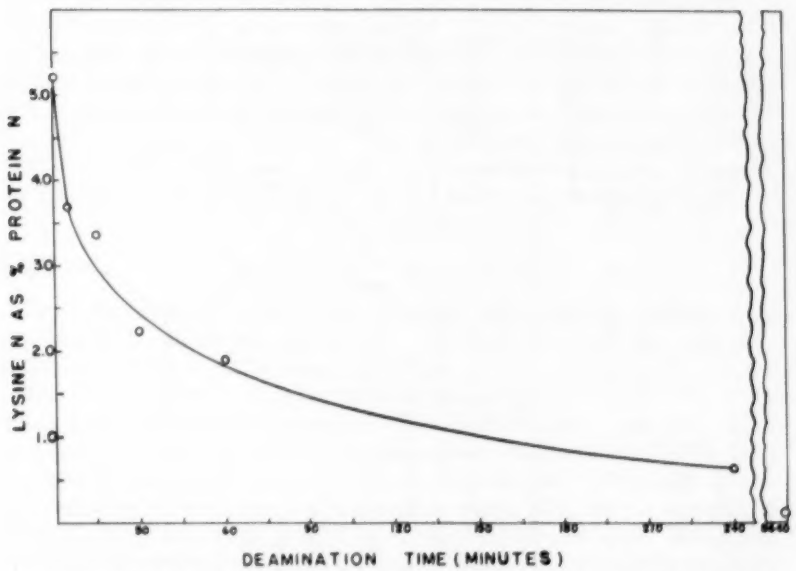

FIGURE 6.—Relation between deamination time and lysine content of collagen.

TABLE III

PARTIAL AMINO ACID ANALYSIS OF COLLAGEN DERIVATIVES

(Amino Acids Changed by Deamination or Decolorizing)

Amino acid	Period of Deamination											
	0 min. %	5 min. %	15 min. %	30 min. %	60 min. %	4 hr. %	12 hr. %	23 hr. %	32 hr. %	50 hr. %	6 days %	23 hr. + Decolorizing %
Lysine + Hydroxylysine	4.9	3.5	3.2	2.1	1.8	0.6	0.5	0.5	0.3	0.3	0.1	0.2
Arginine	8.9	9.1	8.9	8.9	8.5	8.6	8.5	7.7	7.2	7.0	5.8	7.0
Serine	3.8	3.7	3.5	3.8	3.6	3.8	3.6	3.5	3.8	3.5	3.3	3.8
Threonine	2.5	2.3	2.5	2.5	2.3	2.4	2.3	2.3	2.2	2.3	2.2	2.0
Tyrosine	1.1	0.9	0.8	0.7	0.7	0.6	0.6	<0.6	<0.6	<0.6	<0.6	0*
Methionine	1.0	0.9	0.8	0.9	—	—	1.0	0.9	0.9	0.9	1.0	0*

*No spots observed on chromatograms

Lysine.

The Lysine data of Table III is also shown in Figure 1 but recalculated and plotted in terms of lysine nitrogen as a percentage of the total nitrogen. It will be noted that the lysine concentration of the derivatives decreased steadily as the reaction with nitrous acid continued. After six days of treatment with nitrous acid there was practically no lysine present. By examining the paper chromatograms of the hydrolysates of the products, one may readily observe the diminishing intensity of the lysine spot as the deamination proceeds. Excellent separation of lysine was obtained on the chromatograms and no difficulty was experienced with its determination.

The α-amino group of lysine is inactivated by its linkage in the peptide chain, and the decrease in the lysine nitrogen content is attributed entirely to losses in ϵ-amino notrogen. Slightly more than one-fourth of the ϵ-amino groups were removed in the first five minutes of the reaction. It is known that the α-amino groups of the individual amino acids react completely with nitrous acid in five to ten minutes, so the time required for the amino nitrogen in proteins to react could be longer. A reaction with groups other than surface groups requires diffusion of nitrous acid into the inner portion of the fibrous protein matter. The rapid attack of the lysine residues is indicative of their accessibility to nitrous acid. This availability of the terminal amino groups of the lysine side chains in collagen to nitrous acid is stressed by Salo (26).

The attack of the lysine residues as indicated in Table III and Figure 1 is not so fast as that found by Kanagy and Harris (27) who studied the liberation of amino nitrogen as a function of time from wool and collagen in the Van Slyke deamination chamber. These investigators found that more than 40% of the amino nitrogen of collagen was released in five minutes. However, the reaction was carried out at 25° C. and with rather severe agitation.

Arginine.

From the data of Table IV it is evident that arginine residues were not attacked initially by the nitrous acid reagent under the conditions employed. However, in 23 hours a significant drop in arginine content of the collagen had occurred. At the end of 23 hours, approximately 13% of the arginine residues had been attacked. The sample exposed to nitrous acid for 32 hours had only 80% of the original arginine residues intact, while after six days 65% remained. Previous work in this laboratory (18) using column chromatographic methods had shown that about 30% of the arginine of collagen was destroyed in a week of exposure to nitrous acid. Bowes and Kenten (14) observed a loss of 20% when collagen was treated for two days with nitrous acid at room temperature in a manner similar to that described

in this paper. Stubbings and Theis (17) reported a loss as high as 50% after one week of treatment with nitrous acid.

While the course of the reaction of nitrous acid with amino groups is well known, less is known of the reaction with the guanidino group of arginine. Bancroft and Belden (28) showed that guanidine reacts with nitrous acid to give cyanamide and ammonia. Bowes and Kenten (14) postulated that a similar reaction occurs with the guanidine group in proteins, namely,

$$-CH_2-NH-C(=NH)(-NH_2) + HONO \rightarrow -CH_2NHC\equiv N + NH_3$$

According to Kanagy and Harris (27), ammonia is the product of at least one of the reactions of the guanidine nucleus with nitrous acid. These experimenters noted the great similarity of the reaction to its reaction with collagen. Although the α-amino nitrogen of the free amino acid was removed in five minutes, a slow gradual evolution of nitrogen from the guanidine nucleus continued. The α-amino nitrogen atom of arginine in collagen is, of course, combined in the peptide chain and is not free to react.

From the data of Table III it is evident that, under the conditions employed, collagen powder can be treated with nitrous acid for four hours without any significant alteration of the arginine residues, while approximately 90% of the deamination of lysine occur in that time. This length of treatment is preferable to a longer one, if collagen treated with nitrous acid is to be used in studies designed to investigate mechanisms of tanning.

Tyrosine.

It is apparent in Table III that the tyrosine phenolic residues in the collagen chains are attacked by diazotization during treatment with nitrous acid and that this attack proceeds immediately. It was difficult to estimate this amino acid when its concentration was reduced below 0.6%. However, it was apparent from the chromatograms which had higher concentrations of hydrolysate that considerable loss in tyrosine occurred in those derivatives exposed to nitrous acid for one day or longer. Tyrosine spots were absent from the chromatograms of the hydrolysates of collagen deaminated six days or the sample deaminated 23 hours and decolorized by the $KMnO_4$–$NaHSO_3$ treatment.

The tyrosine data on the deaminated derivatives were confirmed by applying Bernhart's modification (23) of the Millon-Weiss colorimetric method to alkali hydrolysates of the deaminated derivatives. The results obtained are presented in Table IV. The results are similar to those found by the

TABLE IV

COLORIMETRIC DETERMINATION OF TYROSINE IN DEAMINATED COLLAGEN DERIVATIVES

Period of Deamination	Tyrosine %
0	1.02
15 min.	0.94
30 min.	0.91
60 min.	0.86
4 hr.	0.68
12 hr.	0.49
23 hr.	0.34
32 hr.	0.27
50 hr.	0.24
6 days	0.19
23 hr. and decolorized	0.18

chromatographic procedure. Eighty % of the tyrosine residues are attacked during six days and they appear to be entirely free and exposed. Philpot and Small (29) observed that diazo derivatives of tyrosine and *p*-cresol are formed with great ease. The one from tyrosine (o-diazodeaminotyrosine) is formed under almost all conditions between 0° and 40° C. In proteins the phenolic group of tyrosine apparently reacts by (*a*) introduction of a nitroso group ortho to the hydroxyl, (*b*) reduction of the nitroso group to an amine, and (*c*) diazotization of the amine group (29).

From the data of Table III and IV it is evident that the decolorizing procedure destroyed most of the tyrosine residues that remained after 23 hours of nitrous acid treatment. It has long been known that tyrosine is readily oxidized by permanganate solution. A number of workers have pointed out that oxidizing agents, particularly if used in excess, will attack the phenolic groups of proteins (30).

Serine, Threonine, and Methionine

The hydroxy amino acid serine is significantly low only in the derivative prepared by a six-day treatment. Although a six-day nitrous acid treatment apparently did not affect threonine, the threonine value of the decolorized sample is significantly low. Methionine represents the only sulfur-containing amino acid found in collagen. The deamination procedure did not affect methionine, but the technique used to decolorize the partially deaminated samples resulted in complete absence of methionine spots from the chromatograms. The methionine content of the decolorized sample was checked by the colorimetric test of McCarthy and Sullivan (24) and found to be negligible.

Methionine may be converted by hydrogen peroxide to either methionine sulfoxide or the next higher oxidation product, methionine sulfone (31, 32). Moran *et al.* (33) have reported partial conversion of the methionine of gliadin and zein to the sulfoxide and sulfone upon treatment of the proteins with chlorine dioxide. The McCarthy-Sullivan test for methionine is negative with the sulfoxide (24) and presumably also with the sulfone. The absence of a positive reaction with the decolorized sample indicates the presence of methionine sulfoxide or sulfone, or both.

It is of interest to note that the solubility of the decolorized sample was considerably increased over that of the parent collagen or of the other deaminated derivatives. While less than 0.5% of either collagen or the deaminated samples dissolved in 24 hours at 25° C. in a phosphate buffer of pH 6.5, 4.0% of the decolorized sample was solubilized under the same conditions. Conversion of a methionine residue to either a sulfone or sulfoxide form would impart a more hydrophylic nature to the product. It is more likely, however, that the increased solubility of the decolorized product is a result of other not readily apparent changes in the over-all structure.

SUMMARY

Two-dimensional paper chromatographic analyses of the amino acids from hydrolysates of the deaminated, partially deaminated, and the decolorized derivatives show that the amino acids, aspartic acid, glutamic acid, glycine, proline, hydroxyproline, leucine, isoleucine, valine, alanine, phenylalanine, and histidine were not attacked by deamination treatment continued for six days or by the decolorizing procedure. The lysine content of the derivatives was shown to decrease progressively. In samples deaminated for periods longer than 12 hour, the arginine concentration was reduced.

The tyrosine concentration progressively decreased and after six days only one-fifth of the original amount remained. The decolorizing treatment also attacked the tyrosine groups.

Methionine was shown to be absent from hydrolysates of the decolorized sample but was unaffected by the deamination procedure. The concentration of serine was slightly reduced in the derivative prepared by reaction with nitrous acid for six days. The threonine value of the sample deaminated 23 hours and then decolorized was significantly lower than that of the parent collagen.

When it is desired to prepare an essentially deaminated collagen derivative for research purposes, it is suggested that a period of four hours be employed for the reaction. In this time, 90% of the lysine residue will have reacted with nitrous acid leaving essentially a deaminated derivative. A four-hour reaction time would cause little or no undesirable destruction of arginine, serine, and threonine, while leaving intact approximately 70% of the tyrosine residues.

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Bonding Film Adhesion Test for Leather Finishes

C. A. Burkart, N. D. Gallagher, and M. B. Neher

Battelle Memorial Institute
505 *King Avenue*
Columbus 1, *Ohio*

ABSTRACT

A test has been developed for measuring the adhesion of finish to leather which gives results independent of other finish properties such as elasticity and flexibility. In this test, a copper strip is bonded to the leather finish by means of a heat-activated phenolic film (or "bonding film"). The force required to remove the copper strip, bonding film, and finish vertically from the leather surface is determined by conventional tensile-test apparatus.

The "Bonding Film Adhesion Test" has been applied to a variety of leathers under different conditions. In general, the finishes on kid leathers show very low adhesion values, cowhide shoe-upper leathers show intermediate values, and upholstery leathers show high values. Within prescribed limits, variation of temperature, pressure, and dwell time during preparation of the specimens do not significantly affect the adhesion values obtained. There are some kinds of finishes to which the presently available kinds of bonding film will not adhere satisfactorily.

Statistical analysis of the test was made on one set of experiments; the experimental error was found to be 0.39 lb. per inch based on 160 determinations. Therefore, the 95% confidence limits are ±0.78 lb. per inch for a single test result.

INTRODUCTION

Adhesion of finishes is an important factor affecting the serviceability of leather. While the degree of adhesion suitable for one kind of leather may vary considerably from that suitable for another kind, it is desirable to know (*a*) what the adhesion values are, and (*b*) whether they are affected by processing variables. Several tests are now in use for this purpose, but they all suffer from one disadvantage or another. In no case do they measure adhesion independently of other finish properties.

Cellophane tape has been widely used to obtain a rough indication of the adhesion of finishes. In this test, cellophane tape is applied to the surface of the leather, usually by hand or thumb pressure. The amount of finish removed when the cellophane tape is pulled from the leather is a measure of finish adhesion. This test, however, is only qualitative since the amount of finish removed from the leather cannot be measured with any great degree of accuracy. Further, the results are influenced by several factors, such as (*a*) the pressure with which the tape is applied to the leather, (*b*) the time elapsing between application and removal of the tape, (*c*) the rate at which the tape is pulled from the leather, and (*d*) whether or not the tape sticks well to the finish.

It appeared highly desirable to develop a method for measuring the adhesion of finish to leather which would give quantitative results and would be as free as possible from other finish properties. This led to the Bonding Film

Adhesion Test which is designed to measure only the force required to pull the finish from the leather in a vertical direction.

BONDING FILM ADHESION TEST

The Bonding Film Adhesion Test was developed as a method of measuring the adhesion of finishes without dependence upon other properties of the finish. In this test the finish is bonded, under slight pressure, to a backing material by means of a heat-activated adhesive film. The leather specimen and the backing material are then pulled apart on a tensile-strength tester. Good results depend on the strength of the adhesive film/finish bond being greater than that of the finish/leather bond.

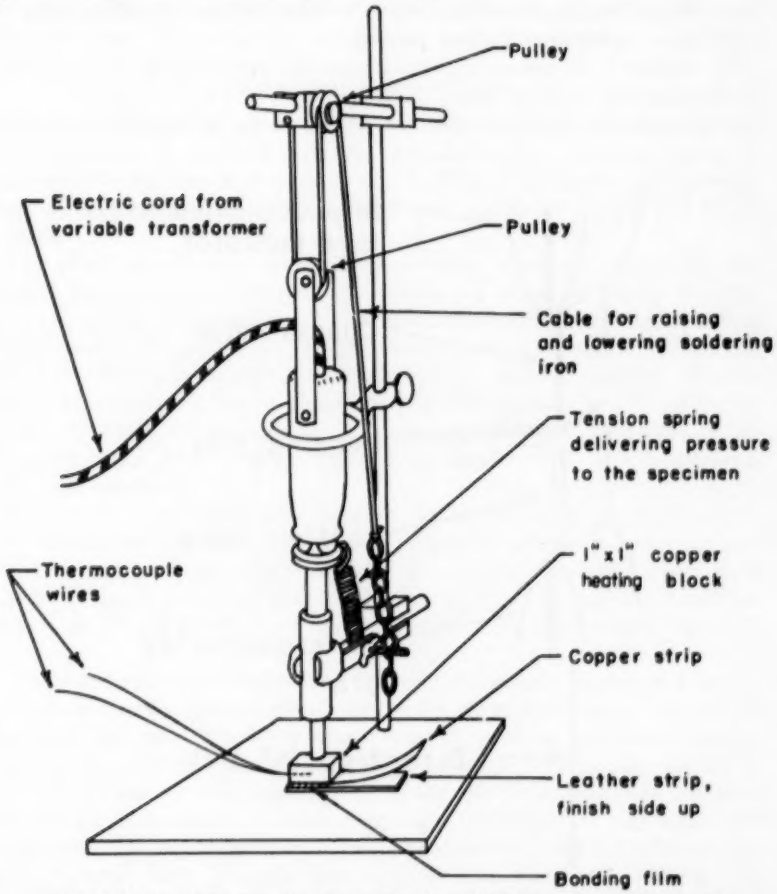

FIGURE 1.—Apparatus for the preparation of bonding film test specimens.

Apparatus.—The apparatus used in the preparation of adhesion test specimens is shown in Figure 1. Obviously there are many modifications which could be made. The essential feature is a source of controlled heat and pressure. The apparatus shown consists of a 100-watt soldering iron equipped with a copper tip one inch square. It is mounted so that pressures as high as 16 psi can be conveniently employed. Temperature is measured by means of a thermocouple and is controlled by use of a variable transformer.

The specimens are pulled on standard tensile-test equipment. A Scott Tester, Model L-6, was used in the work reported here.

Supplies.—The only satisfactory bonding film which has been found is Scotch-Weld Bonding Film Number 583. Sheet copper (8 mil) appears to be the most satisfactory backing material.

The leather to be tested, and the copper, are cut in pieces 1 x 2 inches. The bonding film is cut in pieces 1 x ¾ inches.

It is extremely important that the copper strips be thoroughly cleaned

FIGURE 2.—Sketch showing method of pulling bonding film adhesion test specimens.

before use. This is most readily accomplished by scouring with fine emery cloth. They can be cleaned for reuse by means of various solvents. Chloroform is particularly effective for this purpose.

Procedure.—The leather sample, bonding film, and copper strip are placed together and heated under the specified pressure for the desired length of time. The leather, as shown in Figure 1, is placed on the bottom, finish up. The strip of bonding film is put in place next, with the one-inch width *across* the leather specimen. The copper strip is placed on top.

After cooling to room temperature (25°C.), the samples are aged for at least 4 hours and then pulled on a tensile tester as shown in Figure 2. A pulling rate of 20 inches per minute was used in the work reported here; the effect on adhesion values of varying the rate has not been determined.

RESULTS AND DISCUSSION

The optimum conditions for preparation of the adhesion test specimens vary somewhat for different leathers. Fortunately, however, the optimum conditions for different leathers appear to be fairly close together. In general, these conditions are: 150°–170°C. (block temperature), 8–12 psi, and 10–20 seconds.

The effect on adhesion values of varying the bonding temperature was studied for several leathers, and the results are shown in Figure 3. The minimum temperature which will give satisfactory adherence of the bonding film is around 80° to 100°C. As the temperature is increased further, the adhesion values increase only very slowly at first. In some cases the adhesion values start to rise rapidly at about 170°–180°C. It would seem from Figure 3 that the optimum temperature is in the range of 100° to 140°C. or so. However, much better reproducibility is obtained if 150°–170°C. temperatures are employed.

For pressure and dwell time there do not appear to be optimum values, only minima which must be exceeded. Figure 4 shows the results of two series of tests in which dwell time and pressure were varied. From these graphs, it can be concluded that a dwell time of 10–20 seconds and a pressure of 8–12 psi should be adequate to obtain satisfactory adherence of the bonding film.

The Bonding Film Adhesion Test will give satisfactory results on a variety of leather finishes, such as those obtained with protein, resin, and lacquer emulsion systems. Vinyl top-coated leather, such as upholstery leather, requires temperature approaching 200°C. Higher temperatures should be avoided because of the possibility of fusing the finish and thus obtaining erroneously high results. Lacquer top-coated leathers will not bond even under temperatures as high as 225° C. and cannot be tested by this method at the present time. Further work is being done with other bonding films which may bond to lacquer top coats.

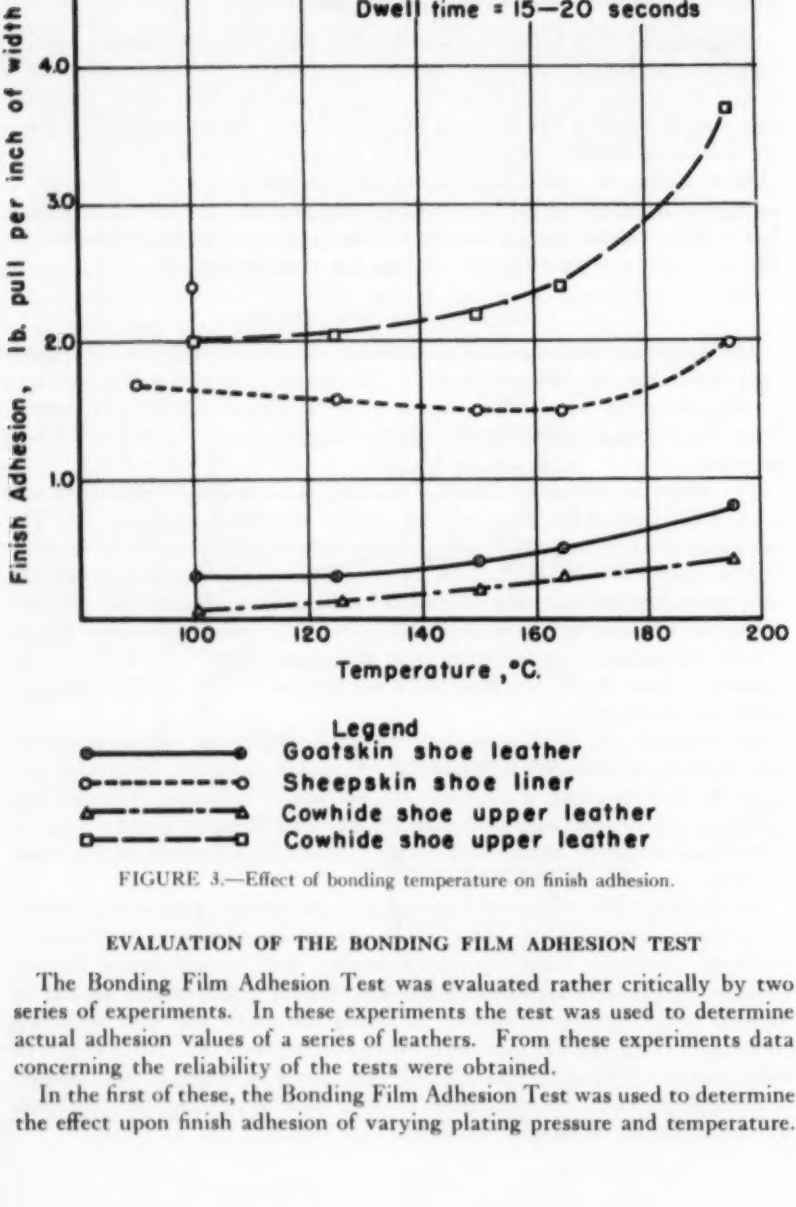

FIGURE 3.—Effect of bonding temperature on finish adhesion.

EVALUATION OF THE BONDING FILM ADHESION TEST

The Bonding Film Adhesion Test was evaluated rather critically by two series of experiments. In these experiments the test was used to determine actual adhesion values of a series of leathers. From these experiments data concerning the reliability of the tests were obtained.

In the first of these, the Bonding Film Adhesion Test was used to determine the effect upon finish adhesion of varying plating pressure and temperature.

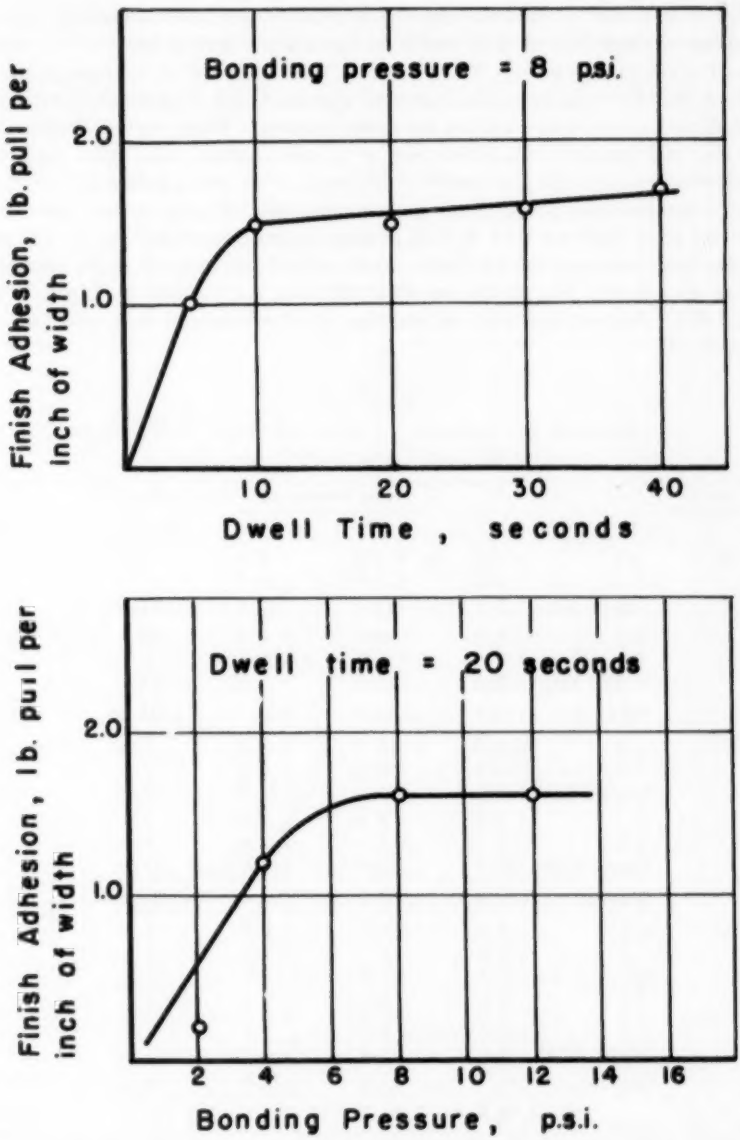

FIGURE 4.—Effect of bonding pressure and dwell time on finish adhesion of cowhide shoe upper leather. Specimens prepared at a blocktemperature of 125°C.

For this series, a chrome-tanned, moderately vegetable-retanned, lightly buffed cowhide leather was used. Samples 2 x 5 inches were cut from the butt areas of two sides. These samples were matched at random in pairs. Each set of two samples was fastened together* and then finished with one of three more-or-less standard finishing systems. These systems comprised sealer and binder coats consisting of a polyacrylate resin plus pigment, followed by (*a*) a lacquer emulsion top coat, (*b*) a polyacrylate top coat, or (*c*) a combination polyacrylate-protein top coat. Plating temperature was varied from 140° to 215° F. and plating pressure from 300 psi to 600 psi. Adhesion measurements were then made in duplicate on each of the two samples in each set. The data from these tests are summarized in Tables I, II, and III. A statistical analysis was run on the data and is summarized in Table IV.

TABLE I

VARIATION OF ADHESION† WITH PLATING CONDITIONS FOR A POLYACRYLATE TOP COAT

Plating Temperature °F.		Plating Pressure, psi				
		300	400	500	600	Total
165		7.0	7.0	6.0	7.0	
		6.5	5.0	6.3	7.3	
	Sample Total	13.5	12.0	12.3	14.3	
		6.0	6.0	6.1	10.5	
		7.4	6.0	5.5	8.2	
	Sample Total	13.4	12.0	11.6	18.7	
	Subtotal	26.9	24.0	23.9	33.0	107.8
190		8.0	8.3	6.3	5.0	
		8.3	8.9	5.5	7.2	
	Sample Total	16.3	17.2	11.8	12.2	
		7.3	8.0	7.2	7.9	
		9.0	7.0	7.4	6.0	
	Sample Total	16.3	15.0	14.6	13.9	
	Subtotal	32.6	32.2	26.4	26.1	117.3
215		7.3	5.5	6.5	8.0	
		8.2	5.5	7.4	7.3	
	Sample Total	15.5	11.0	13.9	15.3	
		8.4	8.2	7.4	8.0	
		7.2	8.0	8.5	7.0	
	Sample Total	15.6	16.2	15.9	15.0	
	Subtotal	31.1	27.2	29.8	30.3	118.4
	Total	90.6	83.4	80.1	89.4	343.5

†Entries are in dial units of Scott Tester, Model L-6. Conversion to pounds is made by multiplying by 0.4. Since the width of the bonding film is 1 inch, this converts to pound-pull per inch of width.

*The assumption of the subsequent analysis of variance would be more nearly fulfilled if these samples had been finished individually.

Probably the most important quantity obtained from this analysis is the pooled error and sample variance. The 95% confidence limits so obtained are $\bar{x} \pm \frac{0.78}{\sqrt{n}}$, where $\bar{x}$ is the average of n determinations.

TABLE II

VARIATION OF ADHESION* WITH PLATING CONDITIONS FOR A POLYACRYLATE–PROTEIN TOP COAT

Plating Temperature °F.		Plating Pressure, psi				Total
		300	400	500	600	
140		3.5	3.5	4.2	4.2	
		3.5	3.0	4.0	4.5	
	Sample Total	7.0	6.5	8.2	8.7	
		7.9	4.3	6.1	5.2	
		5.1	3.5	4.3	4.3	
	Sample Total	13.0	7.8	10.4	9.5	
	Subtotal	20.0	14.3	18.6	18.2	71.1
165		5.5	5.5	7.0	5.5	
		5.5	5.5	5.5	7.2	
	Sample Total	11.0	11.0	12.5	12.7	
		4.5	10.0	7.0	6.0	
		5.5	6.3	5.9	5.5	
	Sample Total	10.0	16.3	12.9	11.5	
	Subtotal	21.0	27.3	25.4	24.2	97.9
190		4.5	6.0	6.0	5.3	
		5.0	5.2	5.3	5.5	
	Sample Total	9.5	11.2	11.3	10.8	
		4.5	5.5	4.5	4.5	
		5.5	5.9	6.0	5.5	
	Sample Total	10.0	11.4	10.5	10.0	
	Subtotal	19.5	22.6	21.8	20.8	84.7
215		8.3	5.0	5.5	6.5	
		7.0	6.0	5.0	6.0	
	Sample Total	15.3	11.0	10.5	12.5	
		7.9	6.5	6.9	6.5	
		7.8	5.5	5.5	6.5	
	Sample Total	15.7	12.0	12.4	13.0	
	Subtotal	31.0	23.0	22.9	25.5	102.4
	Total	91.5	87.2	88.7	88.7	356.1

*Entries are in dial units on Scott Tester, Model L-6. Conversion to pounds is made by multiplying by 0.4.

A number of interesting conclusions can be drawn from Table IV. The low values of F (variance ratio) show clearly that plating pressure has little or no effect on finish adhesion for any of the top coats investigated. For the polyacrylate-protein topcoat and the lacquer-emulsion top coat, plating temperature appears to have a significant effect on finish adhesion. Interaction of plating pressure and temperature does not appear to have a significant effect on any of these finishes.

In the second series of experiments the area-to-area variation of finish adhesion for a number of commercially finished leathers was determined. For this series of experiments samples were taken from the butt, shoulder and belly areas of each side or skin. The adhesion specimens were prepared

TABLE III

VARIATION OF ADHESION* WITH PLATING CONDITIONS FOR A LACQUER EMULSION TOP COAT

Plating Temperature °F.		Plating Pressure, psi				Total
		300	400	500	600	
165		3.5	4.5	7.0	5.4	
		3.5	4.0	.61	5.5	
	Sample Total	7.0	8.5	13.1	10.9	
		2.5	3.2	3.7	6.3	
		2.0	3.6	4.5	6.2	
	Sample Total	4.5	6.8	8.2	12.5	
	Subtotal	11.5	15.3	21.3	23.4	71.5
190		4.6	4.5	6.0	6.0	
		5.7	7.0	6.0	5.0	
	Sample Total	10.3	11.5	12.0	11.0	
		4.0	6.5	6.0	6.5	
		3.5	7.0	5.0	5.5	
	Sample Total	7.5	13.5	11.0	12.0	
	Subtotal	17.8	25.0	23.0	23.0	88.8
215		6.0	6.2	6.5	7.0	
		6.0	7.0	9.2	7.3	
	Sample Total	12.0	13.2	15.7	14.3	
		10.1	7.2	5.5	8.3	
		7.2	7.3	6.0	8.2	
	Sample Total	17.3	14.5	11.5	16.5	
	Subtotal	29.3	27.7	27.2	30.8	115.0
	Total	58.6	68.0	71.5	77.2	275.3

*Entries are in dial units of Scott Tester, Model L-6. Conversion to pounds is made by multiplying by 0.4.

TABLE IV

VARIANCE ANALYSIS OF DATA ON EFFECT OF PLATING CONDITIONS ON FINISH ADHESION

Top Coat	Source of Variation	Sum of Squares	Degrees of Freedom	Mean Square	F	Significance Level
Polyacrylate	Pressure	6.19	3	2.06	0.7	Not significant
	Temperature	4.25	2	2.13	0.7	Not significant
	Interaction	19.08	6	3.18	2.3	Not significant
	Sample	16.65	12	1.39	2.1	.05
	Error	15.65	24	0.65		
Polyacrylate–protein	Pressure	0.61	3	0.20	0.1	Not significant
	Temperature	37.36	3	12.45	5.3	.04
	Interaction	21.25	9	2.36	1.9	Not significant
	Sample	20.12	16	1.26	1.8	.1
	Error	23.09	32	0.72		
Lacquer emulsion	Pressure	15.21	3	5.07	1.9	Not significant
	Temperature	59.96	2	29.98	11.0	.01
	Interaction	16.29	6	2.72	1.8	Not significant
	Sample	25.46	12	2.12	3.4	.01
	Error	14.89	24	0.62		

Pooled Sample and Error Variance—0.966 dial units^2 or 0.154 lb^2/in^2

Pooled Sample and Error Standard Deviation—0.98 dial units or 0.39 lb/in ($\sqrt{\text{Variance}}$)

TABLE V

VARIATION OF FINISH ADHESION* WITH LOCATION OF SAMPLES

Leather Type	Adhesion Results †			
	Butt	Belly	Shoulder	Total
Cowhide shoe upper, heavily buffed	4.6	4.0	3.0	11.6
Cowhide shoe upper, lightly buffed‡	1.4	2.0	1.7	5.1
Cowhide upholstery, corrected grain	4.3	3.0	4.5	11.8
Cowhide upholstery, deep buff	9.9	9.8	11.5	31.2
Sheepskin garment, full grain	1.7	0.8	1.7	4.2
Goatskin glove, full grain	0.6	0.8	1.3	2.7
Total	22.5	20.4	23.7	66.6

*As measured by the Bonding Film Adhesion Test.
†Values recorded are in lb. per in.
‡Semianiline finish; all others pigment finish.

TABLE VI

VARIATION OF FINISH ADHESION WITH LEATHER TYPE AND SAMPLE LOCATION

Source of Variation	Sum of Squares	Degrees of Freedom	Mean Square	F	Significance Level
Location	0.93	2	0.465	1.0	Not significant
Leather type	186.3	5	37.26	82.8	>0.01
Interaction	4.51	10	0.45		

under varying conditions, temperatures 125°–175° C, dwell time 15–30 seconds, and pressure 8–12 psi. For each side or skin, however, all specimens were prepared under the same conditions. The results of this series of experiments are shown in Table V. A variance analysis was run on these data. It is summarized in Table VI.

As expected from the raw data, the leather type is highly significant. Location is not a significant variable. Thus, in using the Bonding Film Adhesion Test, it appears that samples can be taken from any area of the side or skin of leather without making significant differences in adhesion results.

ACKNOWLEDGMENT

We wish to thank the sponsors of the Leather Finishes Research Project of the Tanners' Council of America for their support and for permission for publication. Suggestions and comments by Mr. E. B. Thorstensen, Dr. Ell Dee Compton, Mr. Allen Phillips, Mr. G. Alan Rothschild, Dr. Fred O'Flaherty, and Dr. Robert Lollar have been helpful.

Received March 24, 1956.

Being professional workers, engineers must bear the responsibility for their own development. One of the most effective avenues of professional development—the technical society—relies mainly on engineers for its support. At the same time, industry must recognize its obligation to provide the engineer with opportunities and encouragement to participate in technical society work.

In modern decentralized organizations, engineers tend to find themselves concentrating on specialized problems. A good technical society breaks down the barriers of specialization and brings its members to a better understanding of each other's problems.

—J. S. Alford, *General Electric Review*, **59**, No. 3 and 4, 38 (1956)

REPORT OF THE LEATHER FINISHES COMMITTEE

BERNARD A. ROLL, *Chairman*

The Leather Finishes Committee of the A.L.C.A. offers the following provisional method for testing the resistance of colored leather to bleeding:

K 12

METHOD FOR TESTING RESISTANCE OF COLORED LEATHER TO BLEEDING

1. SCOPE

1.1 This method is intended for use on finished upper leather for shoes or similar end use, or leather articles such as shoes, handbags, etc.

2. APPARATUS

2.1 Air-tight oven at 120° F.

2.2 Four 2-inch squares of bleached cotton sheeting (ASTM Specification D 503–48T). This material contains less than ½% sizing and has a thread count of 186 threads per square inch.

2.3 Two glass plates 6 inches square and ⅛ inch thick.

3. PROCEDURE

3.1 Place the unfinished side of the leather specimen, four inches square, on the center of one of the glass plates. Thoroughly soak the four squares of cotton sheeting in distilled water for five minutes. Stack them on top of each other on the other glass plate. Invert this plate with the cotton cloths and place on top of the other plate holding the leather specimen. The cloths should line up approximately in the center of the leather specimen. The cloth in immediate contact with the leather is marked No. 1; the next cloth, No. 2; the next, No. 3; and the top cloth, No. 4. Place this entire sandwich in an oven at 120° F. for twelve hours (overnight). Remove the glass plates and observe the individual cloths for discoloration.

4. RESULTS

4.1 Migration of Bleeding

Observe each of the four cloths for coloration and report migration as none, 1, 2, 3, or 4, the numbers indicating the cotton cloth that discoloration has reached by migration.

4.2 Hue

Note the predominant hue of the colors which bled on each cloth.

4.3 Intensity

Observe only cloth No. 1 for this purpose. The degree of intensity shall be termed none, slight, moderate, or considerable.

Received May 14, 1956.

OPEN DAYS AT THE BRITISH LEATHER MANUFACTURERS RESEARCH ASSOCIATION

A total of 364 people visited Milton Park during the annual open days of the Research Association, May 2–4.

On May 2, Dr. K. W. Pepper, deputy director, gave the annual lecture to the heavy leather industry on "Improving the Serviceability of Sole leather." Following this, visitors were able to visit the laboratories and see an exhibition showing some of the work in progress.

A similar programme was followed on May 3, following a lecture by Dr. K. G. A. Pankhurst on "Grease in the Light Leather Industry."

On May 4 the visitors were from Government departments and other research and educational institutions. They included a party of post-graduate students from the Royal Holloway College and another party of students of the National Leather-sellers' College.

Government departments represented included the Ministry of Supply, the War Office, the Lord Presidents' Office, the Board of Trade, and the various Commonwealth Scientific Liaison Offices.

—*Leather World*, May 10, 1956.

A three-day Symposium was held by the Leather Technologists' Association (India) at Calcutta on April 22–24, 1956 in conjunction with the All-India Leather Exhibition, which ran from April 22 to May 6. Forty-eight papers were presented covering nearly all phases of chrome leather production, with special reference to the problems of the Indian leather industry. Sixty-three technologists attended from all parts of India.

It would be greatly to the interest of the tanner, and would save him much annoyance, if all hides were imported in a *green* state, that is, merely salted; for when dry, it is very difficult, even for the most experienced, to detect many defects which would impair the quality of the leather into which it is to be converted.

—J. de Fontenelle and F. Malepeyre, *The Arts of Tanning, Currying and Leather-Dressing* (edited from the French by Campbell Morfit), Philadelphia, 1852.

ALCA NEWS

Proceedings, Reports, Notices and News

of the

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Notice to Authors

Authors are requested to introduce each paper submitted to the Journal by an abstract of the article. Articles should also be accompanied by a brief biography of the author(s) for inclusion in "Life Lines", a new feature in the Journal. Unnecessary correspondence will also be avoided if a notation is attached to the copy indicating where the author desires galley proofs to be sent.

GEORGE C. MOSBAUGH

1914—1956

In a tragic accident earlier this year at the Anglo-Canadian Leather Company, Mr. George C. Mosbaugh lost his life in his 42nd year. He was the son of Francis Mosbaugh, a founder of this Association in 1903 and a life member from 1951 until his death in 1952. He became an active member of ALCA in 1938, and was an enthusiastic member until his untimely death.

Mr. Mosbaugh was a graduate of Pratt Institute of Brooklyn where he studied under the late Professor Allen Rogers. His entire working career was spent with the Anglo-Canadian Leather Company, Ltd., at Huntsville, Ontario, Canada, where he succeeded his father as superintendent in 1947. In 1952 he became a director of the company.

He was a member of St. Mary's Roman Catholic Church and a Rotarian, and devoted much of his time to civic endeavors. He leaves his widow, Mrs. Nadine Mosbaugh, and four children, George, Jr., 15; Eric, 13; Mary, 10; and Frank, 4. His tragic passing has brought sorrow to his many friends, both in Canada and the United States.

ALCA's Annual Meeting

The 52nd annual meeting of the Association was held at the Grand Hotel, Mackinac Island, Michigan, June 17 through June 20. The program follows:

SUNDAY, JUNE 17th

2:00 p. m.—Registration
2:00 p. m.—Committee Meetings
6:00 p. m.—Presidential Reception
7:00 p. m.—Smorgasbord

MONDAY MORNING, JUNE 18th

9:00—Registration

TECHNICAL PROGRAM

EDWIN S. FLINN, *Presiding*

9:30—Annual Report
by President Edwin S. Flinn

10:00—FLEXIBILITY GAUGE
by Alfred W. Hopton, Niagara Industrial Laboratories
Discussion: G. L. Kimpel, A. F. Gallun & Sons Corporation

10:30—PROGRESS REPORT ON SOME NEW LEATHER TEST METHODS
by Maynard B. Neher, Battelle Memorial Institute
Discussion: Titus Weaver, Ohio Leather Company

11:00—A LABORATORY METHOD OF TANNING LEATHER FOR THE EVALUATION OF THE EFFECT OF VARIABLES ON LEATHER QUALITY
by Herbert J. Hodus and Robert L. Stubbings, Lehigh University
Discussion: J. T. Chain, Diamond Alkali Company

11:30—NEW CONCEPTS OF RESEARCH FOR THE LEATHER INDUSTRY
by W. D. Johnson and Howard Gottlieb, Bjorksten Research Laboratories
Discussion: W. O. Dawson, Chemtan Company

MONDAY AFTERNOON

Golf Tournament on Grand Hotel Golf Course.

MONDAY EVENING

7:30—Beachcombers Supper Dance in the Casino.

TUESDAY MORNING, JUNE 19th

E. B. THORSTENSEN, *Presiding*

10:00—ZIRCONIUM TANNAGE VII—DIRECT UTILIZATION OF INSOLUBLE ZIRCONIUM DERIVATIVES
by Ian C. Somerville and William J. Rau, Rohm and Haas Company
Discussion: R. G. Henrich, B. D. Eisendrath Tanning Company

10:30—INFLUENCE OF TEMPERATURE ON THE ABSORPTION OF CHROME BY CALF SKIN SQUARES
by Joseph R. Kanagy, National Bureau of Standards
Discussion: H. B. Merrill, B. D. Eisendrath Memorial Laboratory

11:00—NEW DEVELOPMENT IN TANNING WITH ALDEHYDES
by L. Seligsberger and Charles Sadlier, Quartermaster Research and Development Center, U. S. Army
Discussion: H. G. Turley, Rohm and Haas Company

11:30—EVALUATION OF WATER ABSORPTION TESTS FOR SUEDE LEATHER TREATED WITH "QUILON" CHROME COMPLEX
by Albert E. Chouinard, Lowell Technological Institute
Discussion: Mieth Maeser, United Shoe Machinery Corporation

TUESDAY AFTERNOON

FRED O'FLAHERTY, *Presiding*

2:00—STUDIES OF THE CHANGES IN THE NITROGENOUS COMPONENTS OF HIDES AND SKINS I—THE INFLUENCE OF INITIAL PROCESSING OF FRESHLY FLAYED HIDES
by F. L. DeBeukleaer and E. P. Marbach, American Meat Institute Foundation
Discussion: R. M. Lollar, University of Cincinnati

2:30—THE NETWORK STRUCTURE OF ELASTIN IN THE GRAIN LAYER OF CATTLE HIDES
by Edward F. Mellon and Alfred H. Korn, Agricultural Research Service, United States Department of Agriculture
Discussion: R. L. Stubbings, Lehigh University

3:00—SOME NOTES UPON LEATHER QUALITY AND HIDE QUALITY
by Raymond Hauck and Robert M. Lollar, University of Cincinnati
Discussion: A. G. Fleisch, Pfister and Vogel Tanning Company

3:30—PRACTICAL CONSIDERATIONS IN WATERPROOFING LEATHER
by C. W. Mann, Quartermaster Research and Development Center, U. S. Army
Discussion: N. C. Benrud, S. B. Foot Tanning Company

4:00—PHYSICAL PROPERTIES OF LEATHER FATLIQUORED AT DIFFERENT OIL LEVELS
by V. Mattei and W. T. Roddy, University of Cincinnati
Discussion: Bernard Roll, Atlas Refinery, Inc.

4:30—ANNUAL BUSINESS MEETING

TUESDAY EVENING

7:30—ANNUAL BANQUET WITH INSTALLATION OF OFFICERS
Guest Speaker—Mr. Joseph F. Eberle, President, Tanners' Council of America

10:00—Dancing in the Terrace Room

WEDNESDAY MORNING AND AFTERNOON, JUNE 20th

9:30 a.m.—4:30 p.m.—LEATHER MAKING SYMPOSIUM
by the combined Tanners Production Clubs Leather Making Symposium Committee
Subject: CHROME TANNING

Panel:	Chairman	W. R. Lotz, Albert Trostel & Sons Company
	Calf	C. A. Martin, E. Hubschman & Sons, Inc.
	Side	N. C. Benrud, S. B. Foot Tanning Company
	Kid	Harold Bernard, G. Levor & Company
	Sole	J. W. Wolfe, Beardmore & Company, Ltd.
	Sheep	G. F. Shepard, George A. Shepard & Sons
	Upholstery	Gene Rosen, Blanchard Bros. & Lane

PRESIDENT'S REPORT*

By Dr. E. S. Flinn

Fellow Members and Guests: It is a privilege to welcome you this morning to the Fifty-second Annual Meeting of the American Leather Chemists Association. This meeting site was selected in conformance with a long established custom of holding the annual meeting every third year in the

*Presented at the 52nd Annual Meeting, Mackinac Island, Michigan, June 18, 1956.

Mid-West. In this sense, our mid-western members are our hosts today. And we sincerely hope that the location has been sufficiently convenient to permit good attendance by our members from the Canadian side of the border.

Weather permitting, we know you will find the atmosphere at Mackinac Island stimulating. The Convention Committee, under the competent guidance of Mr. Harold R. Miller, has provided a program which, in both technical and social aspects, should be satisfying to all.

Another year has passed by. Again, on behalf of Council in whose hands the stewardship of the Association rests, I would like to give an accounting of the transactions and accomplishments that have taken place. At this time, I would be remiss if I did not call to your attention the contribution each member of Council has made to assure a healthy and vigorous Association. Many times their effort has been spent at a great deal of personal sacrifice. You can show your appreciation by thanking the Council members individually during this meeting.

The Association continues to show a steady growth. At the present time, the total membership, including mutual members, is 1061, of which the active and associate members number 895. This is an increase of 49 members in the past year.

Our financial status is also good. The audited report for the fiscal year 1955 shows that our total income was $34,044 and our total expenses $29,106, leaving a net income for 1955 of $4,938. Of the total revenue, approximately 61% was derived from the operation of the Journal, and this percentage has remained essentially the same for the past ten years. The surplus balance, ending December 31, 1955, amounted to $59,314 and is equivalent to roughly two years' operating expenses. The sound financial condition of the Association reflects the business acumen of principally two men, Mr. Oscar V. Wederbrand, Ways and Means chairman, and Dr. Fred O'Flaherty, Secretary-Treasurer.

While it is good common sense to have a monetary cushion against periods of business readjustments, nevertheless it is equally unwise to have substantial amounts of money that are stagnant and non-productive from a scientific point of view. Last year, I mentioned the action that previous Councils had taken in allocating $1,000 each year to be used in conjunction with the biennial meeting of the International Union of Leather Chemists Societies; on the even year the money being provided to permit an official delegate to attend the biennial conference, and on the odd year the money being awarded to the author of an exceptional technical paper so that such paper could be presented at the International Union Conference. It is strongly urged that future Councils continue this policy which not only effects a closer scientific relationship with our European brethren but can

also stimulate the production of technical papers of fundamental nature from our own membership. Moreover, it is hoped that this surplus balance will continue to be used from time to time to make intelligent extraordinary expenditures that will assist in furthering the scientific development of the leather industry. I am sure that it is not necessary, here, to belabor the obvious by cautioning that any depletion of the surplus balance should be done with the exercise of sound judgement.

It is with a deep sense of sorrow that we record the passing of thirteen of our members since the last annual meeting. These men—Lester H. Armstrong, M. L. Baran, W. H. Barrett, Edwin I. Chalmers, James A. Connelly (a life member), John M. Coyle, W. P. Fritz, Frederick L. Hilbert, George C. Mosbaugh, Curtis S. Peterson, Arthur Sauer, Joseph Stodola (a life member), and our past president and Journal editor Dean Williams—had given staunch support to the Association and will be remembered in many different ways, some for their scientific contributions, some for their vigorous and stimulating discussions on technical matters, and others for their participation in the administration of the affairs of our Association. Their passing is a distinct loss to the American Leather Chemists Association and to the community, and to them we pay homage.

With the passing of our past president Dean Williams, the Association lost not only a man of wise counsel but an editor who for 29 years had piloted the Journal over many a rough course. In the past several years, it had become increasingly apparent that the editorship was an onerous task, extremely time-demanding in nature, and grossly unfair to the individual who assumed this responsibility in addition to his regular full-time position in industry.

Therefore, Council, in a careful examination of the problem, considered and accepted a new concept involving the appointment of an editor-in-chief and a managing editor. The editor-in-chief would be chosen from the membership to serve a three-year term and would not be eligible for re-election. Assisted by an editorial staff, it would be his function to direct the policies of Journal operation, to screen technical articles submitted for publication and to oversee the abstracting of scientific material from other journals. The managing editor, having journalistic experience, would be responsible for all phases of publication from the preparation of the copy for the printer to the reading of the final proofs.

It was believed that, because the term for editor-in-chief was limited to only three years and because the office would carry a certain element of prestige, we could interest members of stature for the position. This belief was amply confirmed when Dr. Henry B. Merrill agreed to become the first Editor-in-Chief. As past president of the Association and as an outstanding contributor of scientific knowledge, Dr. Merrill brings to his new position a

degree of eminence that should make our membership proud. We were fortunate, also, in securing Professor Earl J. Watt of Lowell Technological Institute, Lowell, Mass. as Managing Editor. As Coordinator of Special Services, responsible for publications and public relations at the Institute, his qualifications are obvious.

It must be made clear that this combination editorship arrangement is a new venture, involving increased monetary obligations, and Council is agreed that we must proceed on a probationary basis. Therefore, the assignment is on a one-year trial with the understanding that if everything is satisfactory it will be so continued.

The hallmark of any technical association is its contribution to scientific knowledge peculiar to the industry or industries it serves. The American Leather Chemists Association is no exception. Through its By-Laws, our Association is dedicated to the development and perfecting of methods for the analysis and testing of leathers and all materials used in connection with their manufacture. Over the past three years, each of the technical committees has assumed the huge task of revising its section of the official methods of analysis. This exacting work has finally been completed and every member now has a full set of methods in his possession. I know that you are all aware that these methods are provisional and require an affirmative vote of the active members at an annual meeting to make them official. Consideration for their adoption as official methods will be given during the business meeting to be held on Tuesday, June 19, and it is the definite responsibility of each active member to participate.

The By-Laws further charge the Association to promote the advancement of chemistry and other sciences, especially in regard to their application to problems confronting the leather industry, and to publish the results of such investigations and researches. Here, in this period of extreme competition, the need of research has never been more clearly seen. And it is only through the free exchange between companies of such scientific information that the leather industry can remain strong and show a united front against the outside competitive forces. Therefore, I make a strong plea for the publishing in our Journal of worthwhile researches to be used for a common purpose.

It is research that can spawn a new tanning process or a new leather product. It is gratifying, therefore, to know that Tanners' Council, Leather Industries of America, and organized groups of tanners are spending approximately one-quarter of a million dollars this year in sponsoring research projects at such institutions as Battelle Memorial Institute, Morse Engineering Laboratory (Calif.), Franklin Institute, Lowell Technological Institute, American Meat Institute, and the University of Cincinnati.

All of us have watched a once vigorous sole leather industry slowly shrink due to great inroads made by synthetics. Research, by the development of

the solvent tannage, offers promise in assisting sole leather to regain some of its former position. This can be a typical example of research at work.

During the past year, our technical committees have been reorganized. It is rewarding to observe that many of our younger members have become associated with these committees. Let us hope that more will see fit to participate in this activity which is fundamental to our Association. Also, during the past year, we initiated a new technical committee—the Raw Stock Evaluation Committee. Thus, an important area of the leather industry, long overlooked, will eventually have official methods of analysis developed for it.

It will be recalled that, at the 1955 Annual Meeting, it was voted to make arrangements with the American Society for Testing Materials for a Joint Committee to establish standard methods of testing leather. Such methods would be published by ASTM as joint standards and carry both the ASTM and the ALCA seals. Believed to be of mutual benefit, by providing world-wide recognition to our Association through methods published by ASTM, and by providing the ASTM with methods of analysis for leather that they have long lacked, studies are now being undertaken by a Joint Committee for the development of methods for the physical testing of leather. If this arrangement works out satisfactorily, the chemical testing methods will be similarly treated. Our official methods of the ALCA are being used for the basis of study. We owe a debt of gratitude to Mr. M. Maeser and Mr. E. B. Thorstensen for their participation in initiating this project.

For progress, the leather industry must make full use of the technology available to it. No better way for permitting the practical tanner and the chemist to understand each other's wants is available than to get them together on a common ground. The round-table discussions of practical problems facing the tanner and leather manufacturer, that have been sponsored by the combined production clubs at each of our annual meetings since 1952, have been a most effective way of consolidating these interests. We sincerely hope these practical sessions will become an institution at our meetings. This year, the New England Tanners Production Club, the Delaware Valley Tanners Production Club, and the Tanners Production Club of Wisconsin are joined by the Tanners Production Club of Canada in sponsoring the practical symposium to be held as a full-day chrome tanning session on Wednesday, June 20. We are indebted to the many gentlemen who organized this year's panel discussion, and to them, on behalf of Council, I wish to extend our sincere thanks.

Progress is the result of constant change and of overcoming the static. God willing, may our Association in the coming years be productive and contribute effectively to make the leather industry a dynamic one.

ALCA–ASTM Joint Committee on Leather

Minutes of Meeting May 22, 1956

The meeting was called to order at 10:00 A.M., May 22, 1956, in the Mirror Room of the Hotel Warwick, Philadelphia, by Dr. J. R. Kanagy, Chairman. Each representative present was asked to rise and identify himself by name and affiliation.

The first subject considered was the lineup of subcommittees and sections. The following organization was agreed upon:

Subcommittee 1. Nomenclature, Definitions and Applicability
Chairman: Dr. Robert M. Lollar, Tanners' Council Laboratory

Subcommittee 2. Sampling and Conditions for Physical Testing
Chairman: C. W. Mann, Quartermaster Laboratory, Natick, Mass.

Subcommittee 3. General Physical Testing
Chairman: George A. Butz, Endicott-Johnson Corp.

Sec. 1. Conditioning Leather for Physical Testing
It was agreed that this section would be consolidated with subcommittee No. 2.

Sec. 2. Physical Dimensions
Chairman: Dr. J. Naghski, U. S. Dept. of Agriculture

Sec. 3. Tensile Properties
Chairman: R. Young, International Shoe Co.

Sec. 4. Effect of Water
Chairman: R. G. Ashcraft, Endicott-Johnson Corp.

Sec. 5. Surface Characteristics
Chairman: Ell Dee Compton, Eagle-Ottawa Leather Co.

Sec. 6. Fire Resistance
Chairman: C. Retzsch, Nopco Chemical Co.

Sec. 7. Mechanical Leathers (Miscellaneous Properties)
Chairman: A. N. Compton, E. F. Houghton & Co.

Sec. 8. Deterioration Effects
Chairman: Dr. S. S. Kremen, Leather Research Corp.

Sec. 9. Bond Strength, Belting Leathers
Chairman: Philip G. Rhoads, J. E. Rhoads & Sons

Sec. 10. Miscellaneous Physical Properties
Chairman: A. N. Kay, Howes Leather Co.

Subcommittee 4. Research
Chairman: Dr. M. M. Baldwin, Battelle Memorial Institute

Subcommittee 5. Editorial
Chairman: L. Boor, Philadelphia Quartermaster Depot

Copies of the minutes of the Advisory Committee Meeting held March 13, 1956, had been sent to all members of the Joint Committee. In addition to the information given in these minutes regarding the methods to be considered under each section, the following was agreed upon:

Sec. 7. Mechanical Leathers (Miscellaneous Properties)
Three methods which will be considered are measurement of corrosion, swelling of hydraulic fluid, and friction.

Sec. 8. Deterioration Effects
The determinations which will be considered are perspiration resistance, chemical resistance, washability and dry cleanability.

The proposed by-laws of the Joint Committee were considered at length and several changes were recommended. In the near future copies of the revised proposed by-laws will be sent to all members of the Joint Committee for a letter ballot on their acceptance.

It was agreed that a committee on meeting arrangements was necessary, and B. L. Lewis agreed to accept the chairmanship of this committee. In the future Mr. Lewis will be responsible for making all arrangements for meetings of the Joint Committee. The next meeting of the Joint Committee will be held during the last two weeks of October in New York City.

There was a lengthy discussion regarding whether the methods published under this Joint Committee should employ the English or the metric system. It was agreed that, due to the difficulty of obtaining machines and instruments manufactured according to the metric system in the United States, all methods would employ English units, but that wherever possible an English decimal system would be used.

A luncheon was held in the Junior Mirror Room at 12:30 P. M., after which the meeting was adjourned.

M. H. Battles, *Secretary*,
ALCA–ASTM Joint Committee on Leather

The Perkin Centennial

The 100th anniversary of the dicovery of aniline dyes by Sir William Perkin will be commemorated by a week-long meeting at the Waldorf-Astoria, New York, September 10 – 15, 1956, under the general sponsorship of the American Association of Textile Chemists and Colorists. The American Leather Chemists Association, as one of about 25 participating societies and organizations, will sponsor a portion of one session. Many other parts of the program are of unquestioned interest to anyone concerned with the dyeing of leather. An outline of the tentative program follows. In general, each of the topics listed is sponsored by one or more of the participating societies and will be covered by the presentation of several papers by recognized experts.

MONDAY, SEPT. 10—"INTERNATIONAL DAY"

Morning Topic: "Colorfastness of Textiles"

Luncheon Address, by Sir Robert Robinson of the Society of Dyers and Colourists: "Sir William Perkin—His Life Work"

Afternoon Topic: "Color—the Catalyst of Commerce"

TUESDAY, SEPT. 11

Morning and Afternoon Topics:

"The Application of Colorants to Plastics"
"The Application of Colorants to Drugs"
"The Application of Colorants to Leather" (sponsored by ALCA)
"The Chemical and Physical Nature of Leather Fibers in Relation to Dyeing" by Robert Stubbings, Lehigh University
"Coloring of Leather in Which Penetration Is Desired" by Joseph A. Casnocha, Sandoz Chemical Works
"The Spray or Brush Dyeing of Leather" by C. H. Geister, E. I. duPont de Nemours & Co.
"Dyes and Dyeing Processes for Special Properties of Color" by J. S. Kirk, General Aniline and Film Corp.
"The Application of Colorants to Cosmetics"
"The Application of Colorants to Paper"
"The Physics of Color"
"Color in National Defense"
"The Impact of Synthetic Dyes on Our Economy"

WEDNESDAY, SEPT. 12

Morning and Afternoon Topics:

"The Application of Colorants to Food"
"Pigments and Color Lakes"
"The Psychology of Color"
"Color Harmony from Fashion to Industry"
"The History of Color"

THURSDAY, SEPT. 13

Morning Topics:

"The History of Dyeing"
"The Manufacture of Synthetic Dyes"

Afternoon Topics:

"The Application of Colorants to Textiles"
"Color in the World of Fashion" (stage presentation).

FRIDAY, SEPT. 14

Morning and Afternoon Topic:

"The Chemistry of Synthetic Dyes: a Symposium on a Century of Progress in the Synthesis of Dyes"

Evening: Perkin Medal Award Banquet

Presentation of the Perkin Medal of the American Section of the Society of Chemical Industry to Edgar C. Britton.

SATURDAY, SEPT. 15

Topic: "Theories of Dyeing"

The Official ALCA Representative at the Perkin Centennial is Dr. E. D. Compton, Eagle-Ottawa Leather Co., Grand Haven, Mich. Members of the Association who plan to attend the Centennial may obtain application blanks for hotel reservations and theater tickets by writing to Dr. Compton.

Human skins can be tanned like others. They have more body than cow-skins, and unlike them, are thickest upon the abdomen. Those who have experimented upon this most disagreeable subject, assert that they require a greater number of limings and of exposures to the infusions of bark, and that they swell up a great deal under these operations.

—J. de Fontenelle and F. Malepeyre, *The Arts of Tanning, Currying and Leather-Dressing* (edited from the French by Campbell Morfit), Philadelphia, 1852.

Parchment is the invention of Eumenes, king of Pergamus, in Asiatic Turkey, about 200 years before Christ. It was known in early times, as Pergamena, and was used on account of its durability, for records and valuable manuscripts, and as a substitute for the *papyrus*, or writing-paper of the ancient Egyptians. The finest quality, made from thin and perfect skins, and prepared by very careful manipulation, is called *vellum*.

—J. de Fontenelle and F. Malepeyre, *The Arts of Tanning, Currying and Leather-Dressing* (edited from the French by Campbell Morfit), Philadelphia, 1852.

ABSTRACTS

Paste Drying of Leather. L. Masner. *Kozarstvi*, **5**, 193–94 (1955).—The pelts should be left 0.1–0.2 mm. thicker than normal after splitting. The leather should contain 2.8–3.5% Cr_2O_3 and should be neutralized to pH 5.0–5.5 in the center and 6.5–6.7 on the surface. Retanning with 3–12% of a specially prepared quebracho is useful, but the tannin should not strike through. The leather should contain 25% more fat than normal. In leathers containing 2.8–8.1% total fat, the grain should contain 8.5–19.7% fat. A double fat liquoring is recommended: first, a penetrating fat liquor containing 2% degras and 1.75% of a sulfated oil; then a surface fat liquor with emulsified raw neatsfoot oil. The ratio of oil in the first to the second fat liquor should be 1.65:1, or 3:1 if the leather has been retanned with more than 7% pure tannin on the shaved weight. Penetration of paste is regulated by adding sulfated oil. About 75% of the paste should remain on the plates. Glass plates are most satisfactory. Suede leather and splits are pasted preferably with polymer latexes, which improve buffing characteristics or serve as a base coat. Eukasol Binder S diluted with 30–35% water is recommended. Pig leather is sometimes pasted on the flesh side, and simultaneously given a pigment coat on the grain. The paste should contain about 1% solids. Side leather is dried in 4–8 hr., to 12–15% H_2O. Sometimes the grain is improved by drying on the plates to 18–20% H_2O, followed by free drying. Drying of side leather begins at 50°–60°C. and 35–40% relative humidity in the first section; in the second section 45°–50° and 45–55% relative humidity; in the final section 42°–45° and 60–65% relative humidity. Vegetable upper leather is dried at 40°–45°C., but always below the drop point temperature of the stuffing grease. Suede is dried at 40°–45°C. and 40–60% relative humidity; glazed kid, at not less than 50–60% relative humidity. The flow of air in the dryer should be horizontal, and the direction of flow should be reversed frequently. Continuous 3-shift operation is essential for uniform drying conditions. Gains in surface area of from 6 to 16% are possible, but it is bad practice to strive for the maximum area increase. A sample from the center of a side should stretch at least 20% at a tension of 1 kg. per sq. mm., otherwise the shoemaking becomes very difficult. Data on area changes, based on the initial area as 100, are given below:

Location	Per Cent of Original Area		
	Free-Dried	Free-Dried and Toggled	Pasted
Butt	93.3	93.7	101.9
Shoulder	83.7	95.2	102.9
Belly	89.9	101.0	102.6

The more uniform stretching of pasted leather is obvious. The increased surface area of chrome leather is permanent, but a gain of 8% for vegetable leather was reduced to 4% after storing 3 weeks. L.M.

Sperm Oil in Tanneries. V. Pilc. *Kozarstvi*, **5**, 168–69 (1955).—Cachelot oil from the sperm whale (*Physeter macrocephalus*) contains 66.1% fatty acids, 31.5% unsaponifiable matter, acid value 0.4, iodine value 63, solidifying point 3°–8°C. Cold treatment before sulfating is essential; this raises the iodine value to 73.5 and lowers the solidifying point to −8°C. This product may replace up to 50% of the fish oils normally used for sulfating. Sulfated sperm oil gives good emulsions, but its lubricating value is low. It may be mixed before sulfating with oils that in themselves are unsuitable for sulfating. L.M.

Patent Leather Production. V. Pektor. *Kozarstvi*, **5**, 149–50 (1955).—The best raw materials are light Liniers Frigoificos (10–12 kg. salted weight), young colts, Czechoslovak light goatskins, or Szechuan goatskins. Chrome tannage must be done so as to produce a dense, compact grain. After fat-liquoring, drying, staking, and toggling, the surface is degreased with gasoline. The varnish should be made from refined linseed oil, sp. gr. 0.925–0.935, iodine value 170–80, and unsaponifiable matter not over 1.5%. The oil is boiled till filaments 5 to 7 cm. long are produced. The filaments of the daub coat should be up to 2 cm. long. The varnish is sprayed at 2 atmospheres pressure at not less than 25°C. The first coats are air-dried, and the last coat in a cabinet at 45°–55°C. Final hardening is done either in sunlight or U.V. light. Finally the leather is conditioned 24 hr. at 15°C. and 65% relative humidity to a water content of 14–16%. L.M.

Sole Leathers from Indigenous Tanning Materials. P. V. R. Krishna Reddi, K. A. Joseph, and K. Padmanabhan. *Tanner* (India), **10**, No. 11, 26–8; No. 12, 12 (1956).—"Babul" (*Acacia arabica*) is much used for tanning in northern India. The bark contains 17–21% tannin. The specimen used for experiments contained H_2O 10.0, insolubles 61.5, tannin 20.6, and non-tannins 7.9%; pH of analytical solution 4.0. Tanning tests were made on buffalo hides. Babul alone gave a degree of tannage of only 45 and a yield on the pelt weight of 42%. Further tests were made using 50–75% Babul and 50–25% myrabalans. Best results were obtained with 35% myrabalans and 65% babul. This leather had a degree of tannage of 71% and showed a weight yield of 52%. H.B.M.

Chemical Constitution and Physical Properties of Skins of Some German Fur-Bearing Animals. F. Stather and G. Weinhold. *Ges. Abhandl. Deut. Lederinsts. Freiberg/Sa.*, **12**, 87–91 (1955).—Analytical data and tensile strength measurements are given for skin and hair of 9 species (rabbit, nutria, red fox, polecat, common marten, hamster, pine marten, mole, and cat). The most variable skin component was fat (9–45%). The shrinkage temperatures of the skins, after soaking for 2 days, varied from 61° to 63°C. H.B.M.

Determination and Evaluation of Salts in Natural Tanning Materials, Syntans, and Tanning Liquors. V. Kubelka and A. Blazej. *Chemicki Zvesti*, **9**, 437–44 (1955).—Salts were determined in 18 tanning materials, syntans, and technical liquors by the method of Cheshire, Brown and Holmes (measurement of increased acidity, by titration to pH 6.0, after passing the tannin solution through a cation exchanger). When salts were determined before and after the liquors were used for tanning, a decrease in salt content was found in the cases of certain syntans (Tanigan Extra A, Irgatan AGI, Kortan KN, Kortan Qul and lignin extracts), while others (Syntan K_1, "Naphthalene-difen", and Kortan Extra B) behaved like natural tanning materials. The fixation of salts by pelt was actually higher than indicated by the analytical figures because some salts were brought into the tan liquors by the pelt. L.M.

Salts and Acids in Natural Tanning Materials, Syntans, and Tanning Liquors. V. Kubelka and A. Blazej. *Kozarstvi*, **5**, 235–38 (1955).—To eliminate the error caused by introduction of salts in the pelt, salts were determined by potentiometric titration to pH 6, before and after passage through a cation exchanger, in liquors as prepared and also after detannizing with hide powder by the filter bell method. Materials analyzed included spruce and valonia extracts, Syntan K_1, "Naphthalene-difen", and Kortans KN and Qul. The salts absorbed from some syntans by pelt are supposed to be neutralized acid radicals of the syntan; therefore not all salts removed by cation exchange resins are to be considered as non-tannins. Tanning materials for which the salt content is lowered during tanning contain

cations that form a part of the "tannin". The Cheshire-Brown-Holmes method is suitable for determining salts in vegetable tanning materials, but not in syntans containing "tanning" salts. L.M.

Production of Chrome-Vegetable-Tanned Sole Leather in Czechoslovakia. M. Tomisek, L. Kucka and M. Minarik. *Kozarstvi*, **5**, 208–10, 227–8 (1955).—Processes used in U.S.S.R. and Czechoslovakia are described. Both involve a very light Cr tannage, following a conventional pickle. Vegetable tannage is done in drums (U.S.S.R.) or in suspenders followed by drums (Czechoslovakia). Data are given on tannin content of the sewered liquor from the final drum, with variations in the total amount of tannin given, the composition of the blend, and the number of drums and suspenders. It is concluded that the process is economically feasible only if suspender liquors are used. Analytical data are given on U.S.S.R. and Czech Cr-vegetable sole leathers compared to vegetable sole leather. The thermal stability of the Cr-vegetable leather is higher, but the tear resistance is lower, and the fabrication of insoles is more difficult. The bends are used in the shoe factory without difficulty, but the insoles are not suitable for Goodyear welt shoes. The results do not support the conversion of large sole-leather tanneries to chrome-vegetable tannage. The time of tannage was shortened from 86 to 39 days. L.M.

Tin (II)-Strong Phosphoric Acid. A New Reagent for the Determination of Sulfate by Reduction to Hydrogen Sulfide. T. Kiba, T. Tkagi, Y. Yoshimura, and I. Kishi. *Bull. Chem. Soc. Japan*, **28**, 641–44 (1955) (In English).—A rapid method (1 hr.) is described for determining SO_4 in inorganic sulfates (K alum, Cr alum, and Na, Zn, and $BaSO_4$) with a precision of ± 0.07 mg. in samples containing 15–20 mg. SO_4. The reagent is prepared by dissolving 20 to 80 g. $SnCl_2 \cdot 2\ H_2O$ in 300 g. of strong H_3PO_4 (previously prepared by heating pure, concentrated H_3PO_4 to 300°C. in a current of CO_2) and slowly heating the mixture to 300°C. until colorless, in a current of CO_2, to expel H_2O and HCl. Samples of solid sulfates containing 15–20 mg. SO_4, or aliquots of solutions, are placed in a special distillation apparatus and, in the case of solutions, evaporated to dryness. Then 6 to 10 g. of the Sn^{II}-strong H_3PO_4 reagent is added, and heated slowly to 300° C., sweeping out the H_2S with CO_2 and absorbing it in $Zn(C_6H_5O_6)_2$ solution. Sulfide is determined iodimetrically in the absorbent. So far the method has not been made to work with $MgSO_4$, $CuSO_4$, and other second group sulfates. H.B.M.

Rapid Determination of Sulfur in Organic Compounds by the Sn(II)-Strong Phosphoric Acid Reduction Method. S. Ohashi. *Bull. Chem. Soc. Japan*, **28**, 645–49 (1955) (In English).—The method described in the preceding abstract was applied successfully to the determination of S in "sulfur guanidine" $(NH_2C_6H_4SO_2NHC(NH)NH_2)$, methyl orange, and cystine, and by a modified method in methionine, thiourea, and acetylthiourea. In the modified procedure the sample is treated first with $K_2Cr_2O_7$-strong H_3PO_4, prepared like the Sn^{II}-strong H_3PO_4 reagent, and heated first on the water bath, and then more strongly to decompose the excess dichromate. The Sn^{II}-strong H_3PO_4 reagent is then added, and the determination carried out by the standard procedure. Results agree with the theoretical S content within ± 1%. H.B.M.

Manufacture of Shrunken Grain Leather with Alum-tanned Hides for Gloves. A. Simoncini. *Cuoio, Pelli, Mat. concianti*, **32**, 19 (1956).—Tanners frequently try to obtain shrunken leathers, but the results are not always satisfactory because the operation is made empirically. They think that a good shrinkage is only possible with soft and bodiless hides; on the contrary, a perfect shrunken grain is obtained with more consistent hides of sufficient thickness. The shrunken effect is obtained either by mechanical action or by

chemicals. The hide is formed of fibers and fiber bundles crossing each other in all directions, and the tannage fixes the reticular structure in certain positions. During the tannage, when the hides are repeatedly folded, the fixed wrinkles form on the surface a net-like design. The formation of these wrinkles is facilitated by folding hides with the grain inward, by increasing the concentration of the tanning bath and the speed of the drum, etc. The chemical action which may provoke the shrinking of the leather consists in the fixation of the surface layer, followed by a treatment which may swell the internal layers. In this case the surface fibers do not follow the lengthening of the underlying fibers, the hide undergoes a shrinking and forms on the grain a series of parallel and crossed wrinkles. To achieve this it is necessary to pretan the hides in such a way that only the surface layer is tanned (not more than 25% of the thickness) with very astringent vegetable matters added in the drum in the dry state or, in the case of chrome tannage, with concentrated and highly basic chrome liquors, using a temperature higher than the normal and a rapid turning of the drum. In alum tanning, the shrinkage is better obtained with chemical than with mechanical treatments. An example of the tanning method is the following: the hides are drummed at 20 rpm with 1% NH_4OH and 5% NH_4Cl in 200% water for 1 hr. at 38°C., and, after a short washing with cold water, with 20% of finely powdered sumac leaves for 20 minutes. The shrinkage is done with Tanigan Extra Q 70%, formic acid 12%, water 50% at 38°C. for 3 hr. The last bath is a chrome liquor with 8% Cr_2O_3 of 45% basicity and 200% water applied for 2 hr. It is necessary to basify the bath after this time, because the natural acidity of the hides partially neutralizes the alkalinity of the liquor. The drumming is continued for 2 hr., then the hides are allowed to stand for 24 hr. and neutralized with 3% $NaHCO_3$ in 300% water at 35°C. Fat-liquoring is a very important operation; the shrunken leathers show a rather low resistance to tearing which may be increased by appropriate fat-liquoring, for example, with a blend of 3% egg yolk and 3% neatsfoot oil. Dyeing is done with acid colors, and for the darker colors the use of a mordant (potassium bichromate) may permit a reduction of the dye percentage. G.A.B.

Chromatographical Examination of the Hydrolysis Products of Chestnut Wood. L. Del Pezzo. *Cuoio, Pelli, Mat. concianti*, **32**, 11–17 (1956).—Fresh and exhausted (with boiling water) samples of chestnut wood were hydrolyzed by the following method: After drying, the samples were milled many times to obtain a very fine powder; 1 g. of this powder was treated with 10 ml. of H_2SO_4 (72%) for 4 hr. at room temperature; then, after dilution with 320 ml. water, the solution was boiled under a reflux condenser for 6 hr. From the hydrolysis solution, the excess acid was eliminated by passing through an anionic exchanger (Amberlite IRA-410) until it showed a neutral reaction, the solution was brought to pH 5 with 0.1 *N* acetic acid, and then was concentrated *in vacuo* to a small volume. This fraction was chromatographed on Whatman paper No. 1 using phenol as a solvent and the chromatograms were developed either with $AgNO_3$ (*N* solution + 5*N* NH_4OH) or with aniline phthalate (1.66% phthalic acid + 0.93% aniline in butanol). The hydrolysis of the tanning extract of chestnut wood was made by the same method and the product chromatographed. Examination of the chromatograms shows, in the hydrolyzed fraction of fresh wood, the presence of great amounts of glucose and xylose, together with lesser amounts of mannose, arabinose, fructose, and uronic acids. In the hydrolyzed fraction of the exhausted wood, the same compounds are found with the exception of fructose and uronic acids; in the hydrolyzed fraction of the chestnut extract, the same compounds are found excepting mannose. G.A.B.

Modern Resin Tanning Materials, Their Properties and Uses. R. Heyden and J. Plapper. *Das Leder*, **6**, 215 (1955).—The preparation of urea and melamine methylol compounds is reviewed. These cationic materials can be used only in small proportions with

true tannins because when used alone they condense further and liberate formaldehyde so that the strength of the leather soon drops greatly. Anionic materials with good tanning action can be made from the above compounds by blocking the reactive methylol group through combination with polyvalent phenols such as resorcinol, sodium sulfite or acetate, amino—or hydroxycarbonic or sulfonic acids, etc. The dicyandiamide condensation products of Sellet and Dawson are described. Böhme Fettchemie (Dusseldorf) have a new resin tannin, Drasil II, which can be diluted with hot or cold water to clear solutions, is not sensitive to acid or salts, but is precipitated by vegetable or synthetic tannins. It fills and improves either chrome or vegetable leather. A white, wash-fast, glove leather can be made by tanning sheepskin with 3–5% of this resin, 6–8% of Lorikal (an anionic fat product) and 1–2% of alum. Drasil I is a condensed anionic resin tannin that can be diluted in any proportion with water to form stable solutions. It is not sensitive to salt but precipitates below pH 6.0. It has a filling action when used as a pretan for chrome leather. The classical urea and melamine resins are fixed to hide and leather mainly by increase in molecular size, but the dicyandiamide resins are taken up by hide as cationic or anionic substances in the same way as other higher molecular ionic materials such as dyes or emulsifiers. They also can be deposited by precipitation with salts, acids, or oppositely charged materials. I.D.C.

The Fundamentals of Tanning with Synthetic Tanning Materials. An Attempted Classification of Organic Tannins. A. Küntzel. *Das Leder*, **6**, 200 (1955).—The following classification is proposed:

I. Aromatic tannins
  A. With cationic charged groups
    1. Cationic tanning materials (aniline derivatives)
    2. Amphotannins
      a) Synthetic phenolic amphotannins
      b) Amphoterized vegetable tannins
  B. Without substantivity
    1. Condensed naphthalene sulfoacids
    2. Phenolic auxiliary tannins
    3. Lignin sulfoacids
  C. With substantivity
    1. Polysulfonamides
    2. Phenolic exchange tannins
    3. Vegetable tannins

II. Aliphatic tannins
  A. Natural fats
  B. Polymerizates
    1. Train oil polymerizates
    2. Vinyl polymerizates
      a) Prepolymerizates (du Pont G 942)
      b) Polymerization in hide
        (1) Water-free
        (2) In aqueous emulsion
  C. Diepoxide, epichlorhydrin, etc.
  D. Paraffin sulfochloride (Immergan, Skelt)
  E. Alky sulfate (Lorikal)

III. Resin tannins
  A. Dicyandiamide-formaldehyde resins
    1. Modified dicyandiamide resins (Chemtan R6, Retingan R6, Drasils)
    2. Simple dicyandiamide resins (Chemtan R33)
  B. Condensation tannins (urea or melamine methylol compounds)
  C. Polyaddition compounds (di-isocyanate, Gerbstoff H)

IV. Tanning Aldehydes and Quinone
  A. Mixed unsaturated dialdehydes (chamois tanning)
  B. Glyoxal, methylglyoxal
  C. Formaldehyde in combination with many tannins of the other groups
  D. Quinones

All tannins with high astringency, good filling action, and capable of being used alone for tanning are aromatic tannins. The benzene ring plays an important role in the affinity for hide substance as it permits formation of dipoles. The phenolic hydroxyl is not as important in tanning as it was formerly thought to be. The tannins with dipole groups are classed as substantive or direct combining syntans. Non-substantive tannins have no important dipolar groups and combine with hide by salt bridges between the basic groups of hide and sulfo groups of the syntan. The dividing line between these groups is not sharp and depends among other things on the degree of sulfonation. Lignin recently has been modified to form a substantive tannin. The substantive class tan is neutral or slightly acid solutions, but the non-substantive tannins require acid to develop their tanning power. The amphotans are a new class of tannins. The tannins of the second main group are mainly hydrocarbons with groups (usually end groups) containing oxygen and sulfur. The only nonsynthetic members are the natural fats. In chamois tanning the oil polymerizes, coats, and separates the hide fibers. Treatment of leather with prepolymerized resin is usually referred to as impregnation. Treatment in an aqueous emulsion with a catalyst so that some polymerization occurs within the leather is referred to by Batzer as polymerization tannage. The principal use of tannins of the aliphatic class is to make soft, supple leathers such as chamois and glove. Because their tanning ability is limited, they have restricted uses alone for tanning leather, but they are used in combination with a wide variety of more active tanning materials. Tannins of the third main group are useful for filling and increasing cutting value of flat, empty chrome leathers without changing their chrome character. They are important now when cattle hides are being used increasingly for upper leather instead of sole leather. The oldest group of resin tannins, the methylol compounds of urea, melamine and recently furans, do not tan but are used to fill between the fibers. They are always used with formaldehyde. The dicyandiamide-formaldehyde condensates are prepared so that a slight change in pH will precipitate them. The tannins of the fourth main group are all synthetic. They have great tanning power but no filling ability, and therefore are used with other materials. I.D.C.

Tannins in Aniline Printing Inks. A. Angelini. *Chimica* (Milan), **12**, 51 (1956).—For the preparation of printing inks, tannins are added to the solution of a basic dye in an organic solvent in the proportion of 1 mole dye to about 4 moles tannin; the tannin-dye lakes have a high coloring power and brilliancy, and are very resistant to light and rubbing. Several tests to find out what type of tannin is most suitable for this purpose show that the hydrolyzable pyrogallol tannins are more useful than the catechol tannins, because the flavone and anthocyanine derivatives do not give good complexes with the dyes. The examination of the tannins was made by paper chromatography using a mixture of butanol-acetic acid-water as a solvent, and $FeCl_3$, $AgNO_3$ and diazotized sulfanylic acid as reagents for developing the spots. G.A.B.

The Chemical Principles of Vegetable Tannage. T. White. *J. Soc. Leather Trades' Chemists*, **40**, 78 (1956).—A review is presented of the various published ideas about the manner in which vegetable tannins react or combine with hide to make leather. The present knowledge of the nature of collagen and of vegetable tannin extracts is briefly surveyed. The potential sites of combination present in the reacting system are identified and the physicochemical aspects of the system which may contribute to the leathering effect are discussed. The paper reviews, in the light of present knowledge, suggestions that vegetable tanning be attributed (*a*) to electrical neutralization of oppositely charged colloids, (*b*) to coordinate bonding, with hydrogen bonding as a particular mechanism, (*c*) to dehydration and elimination of bound water, (*d*) to undefined adsorption effects, (*e*) to oxidation-reduction processes involving reaction of tannin molecules as quinones, and (*f*) to physical deposition of insoluble matter. Primary fixation is attributed to coordinate or hydrogen bonding with physical deposition playing a part which has not been quantitatively assessed. There are 83 references. J.M.C.

The Resistance of Leather to Enzymatic Degradation by Trypsin. F. Stather, S. Walther, and U. Stather. *Das Leder*, **7**, 13 (1956).—The main criteria of a true tannage are shrinkage temperature (T_s), hot-water stability, resistance to enzymatic decomposition, and certain physical changes in the hide fiber texture. Little use has been made of the enzyme test for studies on tannage, and the relation of this test to others is not sufficiently clear. Although only the surface of collagen and leather is attacked by enzymes, hide powder was used in preliminary work with the hope of obtaining more nearly quantitative results. In contrast to previous workers, no buffer solution was added to regulate pH. Enzyme activity may have been affected by chance pH changes, but the fundamental character of the leather was not changed. For this work a chemically completely untreated hide powder was used. It was made from the middle split of an unlimed cowhide, and about 90% consisted of particles of 0.5–2 mm. The enzyme material was a clear water-soluble trypsin preparation with a strength of 190,000 Fuld-Gross units. One g. portions of dry hide powder, or leather containing an equivalent amount of hide powder, were weighed into 150 ml. flasks and digested for 2 hr. at 37°C. with 100 ml. portions of 5% (hide powder basis) enzyme solution (0.5% solution). Nitrogen was then determined microanalytically in the filtrate. A blank was run with water instead of trypsin, and a correction was also made for nitrogen in the enzyme preparation. Trypsin number is the amount of dissolved hide substance as percentage of the amount taken. The vegetable and syntan tanning was done by rotating in a bottle for 24 hr. 7.5 g. of hide powder (soaked 1 hr. in 75 ml. of water) with 225 ml. of centrifuged tan solution containing 30%, 60% or 160% of pure tannin based on the hide powder weight. The tanned powder was then washed continuously for 12 hr. with 10 l. of water. The resistance of vegetable-tanned leather to trypsin increased with increasing tannage and was practically complete if the degree of tannage was 25 or more. Different results by other workers may have been the result either of insufficient tanning or of detanning by the buffer solution. In the present work there may have been a lowering of enzyme activity because the solutions were not buffered. Syntan leathers were quite resistant to trypsin if completely tanned. Even a weak chrome tannage made the leather almost completely resistant. Alum-tanned leather had little resistance, and satisfactory resistance was never obtained with lignin, nor with even 1% fixed formaldehyde. The work was repeated using pieces of skin instead of hide powder. A delimed sheepskin was dehydrated with acetone, then cut into 5x5 mm. squares. For each determination a sample of skin or leather containing 1 g. of hide substance was digested with 100 ml. of trypsin solution for 4 hr. at 50°, shaking by hand every 15 min. The trypsin number was determined as above. The leathers were made by tanning 7.5 g. portions of skin in 300 ml. of solution. Hot-water stability was determined by heating an amount of leather containing 1 g. of hide substance in 80 ml. of hot water for 7 hr. in a boiling water bath, shaking at

15-min. intervals, then filtering and determining nitrogen in the filtrate. Hot-water stability is 100 minus the number of grams of hide substance dissolved from 100 g. of leather. The trypsin number of untanned hide powder was 74.6 and of the squares 68.4, but all tanned squares were more resistant to trypsin than the tanned hide powders. The results with squares are shown in the following table (slightly abbreviated):

Tanning Material	Tannin Used in Tanning as % of Skin	Degree of Tannage or % Cr_2O_3, Al_2O_3 or HCHO in Leather	End pH of Enzyme Solution	Trypsin Number %	Shrinkage Temperature, T_s	Hot-Water Stability %
None	—	—	6.6	68.4	60	32
Quebracho	15.0	14.0	6.0	4.7	72	48
"	30.0	21.7	6.2	0.9	76	55
Chestnut wood	15.0	11.9	5.8	3.0	68	59
"	30.0	23.4	5.7	1.5	71	72
Wattle	15.0	14.8	6.0	2.5	74	56
"	30.0	25.0	6.0	0.2	75	78
Valonia	15.0	9.1	5.8	5.2	67	49
"	30.0	19.0	5.7	0.4	69	73
Tanigan Extra A	15.0	10.7	5.9	5.0	67	41
"	30.0	17.1	6.0	0.3	68	51
Tellutan EZE	15.0	8.7	6.0	8.0	66	37
"	30.0	14.3	5.7	3.7	68	52
Novaltan V	7.5	2.8	5.9	5.5	52	17
"	15.0	10.3	5.1	0.0	53	34
Lignin ext.	15.0	9.7	5.9	15.1	62	43
"	30.0	15.6	5.9	15.0	63	57
Basochrom	1.5	1.4	6.1	0.7	82	72
"	2.5	2.3	6.1	0.0	90	88
Aluminum alum	2.0	1.2	5.8	16.0	65	14
"	4.0	1.5	5.8	10.6	66	6
Formaldehyde	5.0	0.3	6.0	8	77	63
"	10.0	0.5	6.2	6	78	71

A degree of tannage of about 20 is sufficient to render the leather stable to trypsin. Syntans were nearly equal to vegetable tannins, but lignin, alum, and formaldehyde leathers were never resistant to attack by trypsin. Chrome leather was completely stable. Usually trypsin resistance was satisfactory if the shrinkage temperature was 68°–69° or over, but formaldehyde leather was an exception. There was little relation between trypsin number and hot-water stability I.D.C.

Electron Microscope Studies of the Processes In Leather Manufacture. I. A Preliminary Study of the Structure of Leather. D. Burton, R. Reed and M. J. Wood. *J. Soc. Leather Trades' Chemists*, **40**, 91 (1956).—This is a preliminary report, of a general and exploratory nature, of electron microscope studies of various processes used in leather manufacture. Chrome calf, zirconium calf, chrome-retan cowhide, vegetable calf, vegetable goat, vegetable sole leather, kip tanned with vegetable followed by chrome, and oil-dressed sheepskin were examined. Chrome tannage is shown to be milder and more uniform in its effect on collagen fibers than is zirconium tannage. Compared to straight

chrome leather, chrome-retan fibrils are slightly fuller, and the spaces between are filled with large, dense particles. Vegetable tannage produces marked modification of the collagen fibrils. Here too the presence of large dense particles between fibrils is evident. It is suggested that these are formed by aggregation of smaller particles. Fullness in leathers appears to be associated with broad fibrils of collagen and the presence of dense particles in interfibrillar spaces. J.M.C.

Comparison of Methods of Degreasing Employing Anionic and Non-Ionic Accessory Agents. R. F. Innes and K. G. A. Pankhurst. *J. Soc. Leather Trades' Chemists*, **40**, 99 (1956).—The degreasing of pickled sheep pelt was investigated. The effect of adding non-ionic (long chain phenol-ethylene oxide type) and anionic (sulfated amyl oleate) surface-active agents in the normal solvent (high boiling hydrocarbon mixture) degreasing procedure, as well as their use in an aqueous emulsifying process with wet pelts, was evaluated. Both laboratory and full-scale tannery degreasing experiments were made. All results indicated the superiority of the solvent method compared with the emulsion technique. The non-ionic agent is more efficient as an accessory agent in the solvent procedure than is an anionic one. With neither accessory is the process satisfactory when the skins contain intact fat cells. J.M.C.

Amino Acid Composition of Elastoidin. M. Damodaran, C. Sivaraman and R. S. Dhavalikar. *Biochem J.*, **62**, 621 (1956).—Elastoidin fibers were isolated from the dorsal fin of a shark. Fresh skin was split in two halves and soaked 48 hours in changes of water with toluene as a preservative. The fin-rays were pulled from the fleshy portion of the fin and washed free from adhering tissue. The isolated fibers were treated with a bicarbonate-buffered trypsin solution at 37°C. for 48 hr. Decanting of the enzyme solution was followed by repeated washing. For analysis the fibers were macerated in a Waring Blendor with water of 0°C. The disintegrated material was filtered, washed with acetone, and extracted with petroleum ether in a Soxhlet. Nitrogen, corrected for an ash content of 0.73%, was 17.52% on a dry basis. Amino acid analyses of hydrolyzed aliquots were made with Dowex-50 columns. The amino acid composition exhibits the main features considered characteristic of mammalian collagens, i.e., 32% of the total residues is glycine, the proline and hydroxyproline content is fairly high, and the relative distribution of total basic and total acidic amino acids is the same as found in collagen. The hydroxyproline value (5.34% on a nitrogen basis) is somewhat less than that of mammalian collagen. A tyrosine value of 7.15% (by weight) and cystine of 0.35% are striking deviations. Electron microscope examination of fibers fixed with formalin gave band spacings between 750 and 880 A. The shrink temperature was 63°–64°C., and a somewhat reversible shrinkage was observed. J.M.C.

Prevention of Running Grain in Chrome Leather. L. Masner. *Kozarstvi*, **5**, 152–53 (1955).—The cause of running grain is often too long a soak. Liming with 3–5% of Na_2S (60%) and 2–6% CaO is recommended. Sometimes addition of 3–5% NaCl is useful. When liming with a high proportion of CaO, at temperatures above 15°C., the time of liming should be shortened. The flanks should not be fleshed clean. The pelts should be delimed without delay and bated as little as possible. Bating should start at pH 6–7, and the pH should not be raised to 8–9 until deliming is complete, to prevent overbating and production of loose flanks. Fuller bellies are obtained by replacing HCl by H_2SO_4 in pickling. The float during chrome tanning should not exceed 100%, and the basicity should not be over 40%. Some flesh should be left on the bellies after shaving. In fat-liquoring, calf leather should receive 0.8–1.0% pure fat (never over 1.4%). It is better to fat-liquor less, and add the necessary neutral fat or an ammonium soap to the flesh sides of the butts before first drying. When using sulfated oils only, the flanks are easily over-greased.

Buffing of the bellies should always be done from tail to head—never from spine to belly. The bellies should be glazed only once, even if the rest of the leather gets 2 glazings. It is also possible to set the grain before chrome tanning by metaphosphates (Calgon). In the first stages of drying a high relative humidity should be maintained, followed by a rapid drying at 45°–50°C. L.M.

The Tanning of Sole Leather. M. Tomisek. *Kozarstvi*, **5**, 63–64 (1955).—After a review of sole leather tanning in different countries, analyses are given for British, U.S.A., and Czech leathers. Czech bends contain ash 1.7, fat 1.0, water solubles 12.5, hide substance 41.4, fixed tannin 30.8%; pH of water solubles 4.3; difference figure 0.7; water absorption in 2 hr. 31.8%. L.M.

Quality of Sole Leathers. M. Spicka. *Kozarstvi*, **5**, 24–8 (1955).—The quality of Czechoslovak sole leathers is discussed. The use of high percentages of syntans, sulfited quebracho, and sulfited spruce bark extract, as well as oiling with sulfated oils, raises the amount of SO_3H groups in the leather, which leads to high water absorption. Experimental tannages (results to be published later) are described. These include replacement of sulfited quebracho by wattle, elimination of sulfite in the extraction of spruce, and reduction of proportion of syntans from 30 to 20%. L.M.

Impregnation of Vegetable-Tanned Leather. I. Binko. *Kozarstvi*, **5**, 105 (1955).—A filler formula, taken from an encyclopedia published in 1798, is given. A mixture of nut oil 8, linseed oil 24, $ZnSO_4$ 8, and $Pb(C_9H_9O_2)_2$ 8 parts is boiled slowly, and sedimented. Colophony 6, black tar pitch 3, wood tar 1.5, and turpentine 1.5 parts are added, and the mixture is heated again. After decanting, 4 parts turpentine are added. The mixture is applied to dry leather. The author thinks that 160 years have added little to our knowledge of impregnating leather. L.M.

Electrostatic Forces in the Finishing of Furs. R. Vrbacky and V. Vrbacka. *Kozarstvi*, **5**, 14–19 (1955).—The effect of static electricity in the finishing of furs is described. The most serious difficulties arise in the finishing of rabbit skins. The felting tendency of these furs, formerly attributed to the nature of the skins, is the result. The origin and prevention of electrostatic potential are described. An apparatus for measuring the e.s. potential of furs, after 10 experimental frictions (U_{10}) is described. For a raw, dried rabbit skin, U_{10} is 560 V. on the spine, 1210 V. on the cheek, and 730 V. on the sides. The increase is due to higher hair density and changes in hair direction. Felting tendency is greater in the places showing higher U_{10} values. After tannage with HCHO and alum, U_{10} (spine) was 71; after Cr tannage, 78; after a pseudotannage (pickling with 4% H_2SO_4), 7; and after HCHO tannage, 533 V. The low values are due to the presence of salts. Dyeing changes the U_{10} values of the above leathers to 507, 445, 458 and 653 V. respectively. Mordants increase U_{10} values in the order: $K_2Cr_2O_7$, $FeSO_4$, $CuSO_4$, no mordant. U_{10} values are unaffected by quantity of dye (Ursols) and by high grease content, and are lowered by dyeing with aniline black. Treatment of dyed furs with salt solutions (NaCl or $AlCl_3$) lowers U_{10} values. The range of U_{10} values for no felting tendency is 300–400 V., moderate felting tendency 300–600 V., and high felting tendency 500–1000 V. L.M.

Gelatin from "Acid-limed" Fleshings. A. Bycichin, J. Lacnak, and B. Nemec. *Kozarstvi*, **5**, 146–48 (1955).—Flesh splits of limed steer hides and hog skins were washed 18 hr. in water at 14°C., and "limed" in 200% of a 2% solution of acetic acid at 20°C. After 6–8 days the solution, which then contained 0.8–0.9% acetic acid, was replaced by a fresh one. The splits were washed after 40 and 200 days. The gelatin leaches out slower than from normally limed stock. The physical and photographic properties of "acid-limed"

gelatin are superior. The iso-electric point is pH 7, instead of pH 5 for normal gelatin. The acid gelatin is suitable for color photography. The disadvantage of the acid process is the need for preliminary degreasing. Total processing time is about the same. L.M.

Lime Slaking for the Liming of Hides. B. Plechac. *Kozarstvi*, **5**, 150–52, 170–72 (1955).—Mechanical contrivances for slaking lime are described. Use of 200–250 parts of water per 100 CaO is recommended. Circumstances favorable for slaking include small lump size, high purity, and soft water of a higher temperature. The slaked lime should "ripen" for at least 10 days. Sedimentation and utilization of a pure lime cream are recommended. Slaking in a rotating tannery drum is advocated. L.M.

The Chromatography of Vegetable Tannins. Z. Kotasek. *Kozarstvi*, **5**, 95–97, 114–17 (1955).—The chromatography of extracts of spruce bark (*Picea excelsa*) is described. Whatman W-1 and Schleicher & Schull 2043 papers were used, with the following solvent mixtures: (*a*) n-butanol - acetic acid - water (4 : 1 : 5); (*b*) *a* + 5–10% ethylene glycol; (*c*) butanol -pyridine - saturated NaCl solution (1 : 1 : 2); (*d*) ethyl acetate - acetic acid - H_2O - ethanol (3 : 1 : 3 : 0.5); (*e*) same as *d* in the proportions 2 : 1 : 2 : 0.33. The tannins of spruce bark contain at least 6 fluorescent compounds: 3 blue, 2 yellow, 1 brown-blue. The yellow spot with a high R_f value corresponds to a component of quebracho. Alkali extracts of spruce bark give no fluorescence even after removal of cations by ion exchange. Eight components give reactions of polyphenols. The fluorescent spots are connected by fluorescent blue bands. The sugars in the spruce non-tannins were chromatographed by *a*, *c*, pyridine - ethyl acetate - water (2 : 1 : 2), and butanol - pyridine - water (12 : 8 : 7). R_f values in *a* were rather low. Comparison with chemically pure sugars was made after removing anions and cations from the non-tannin solution. The most important aldohexose component is glucose; arabinose and perhaps xylose and ribose are also present, as well as some decomposed uronic acids. This indicates the presence of pectins, which affect tanning with spruce adversely because of their high viscosity. Other pentoses, ketohexoses, ditri- and polysaccharides, as well as sugars bound as glycosides or polyphenols will be discussed in a later paper. The pharmaceutical significance of tannins and flavanoids is mentioned, e.g., protection from irradiation by radio-isotopes. Other applications of chromatography in the leather industry (lime liquors) are indicated. L.M.

New Analytical Methods in Tanneries. A. Stehlik. *Kozarstvi*, **5**, 35–7 (1955).—The Bergmann or the Schooper apparatus for determining permeability of leather to air has been replaced by Fodor's apparatus (not described). The "Gost" (USSR) apparatus for determining permeability to water is being tested. The hydrothermal test for determining the stability of vegetable-tanned leather has been introduced. Replacement of hide powder by some better standardized polyamide, in tannin analysis, is advocated. A new method for the determination of organically bound SO_3 in sulfated oils (see *Chem. Abstr.*, **49**, 8615*i*) gives better results if an excess of H_2SO_4 is added, and back-titrated after removing CO_2 by boiling. Czechoslovak sulfated oils contain 2.3–5.0% organically combined SO_3. Sulfated oxyparaffine contains only 0.9%. An analytical evaluation of sulfochlorides has been worked out. S and Cl are determined in the Grote-Krekeler combustion apparatus. The combustion products are absorbed in alkaline H_2O_2 solution. The Cl content is always higher than the theoretical Cl : SO_3 relationship, and therefore Cl is bound also by addition or substitution. A good insight into the sulfochlorination process is given by determining the saponification value. The SO_2Cl content calculated from the equation:

$$\cdot SO_2Cl + 2\ KOH = \cdot SO_3K + KCl + H_2O$$

gives the same results as the polarographic determination of Majranovskij and Nejman (see

Chem. Abstr., **46**, 28*e*). For the polarographic examination of sulfochlorides used in tanning, cyclohexanone is a suitable solvent. A good mixture consists of 15 ml. 5 *N* $Ca(NO_3)_2$, 15 ml. cyclohexanone and 20 ml. ethanol. A cyclohexanone solution of the sulfochloride is added to this solution. The half-wave depolarization potential is near zero (to *N* calomel electrode). There is a linear relationship between sulfochloride concentration and the height of the wave, unless the proportion of solvents is changed. A pure *p*-toluene sulfochloride was used to evaluate the curve by the method of standard increments. The determination is possible up to a concentration of 0.001 *M* sulfochloride. Higher concentrations give maxima on the curves, which can be depressed only with difficulty. A reverse exposition of polaragrams, from a higher to a lower negative potential is advantageous; the wave develops better. A distinct Cl^- wave develops after the sulfochloride wave, permitting their simultaneous determination. This method can be used to determine the degree and rate of hydrolysis of the sulfochloride. The polarographic method may be used in controlling tannage by sulfochloride. L.M.

Principles of Vegetable Tanning. M. Tomisek. *Kozarstvi*, **5**, 144–6 (1955).—A review. L.M.

Relation of Upper Leather Finishes to the Finishing of Shoes. L. Masner. *Kozarstvi*, **5**, 103 (1955).—A review. The temperature of ironing shoes should not exceed 80°C. in the case of resin-finished leathers. L.M.

A Review of Actual, and a Preview of Future, Finishing of Leather. V. Pektor. *Kozarstvi*, **5**, 45–6 (1955).—After a brief review of current processes, the necessity of using structurally soft synthetic resins is stressed. The Soviet method of utilizing polymethacrylates with addition of plasticisers before polymerization has also been tested. Several types of dispersions of synthetic resins of different elasticity and hardness are necessary for the successful finishing of shoe upper leathers. In Czechoslovakia a dispersion of a copolymer of 80% butyl methacrylate and 20% butyl acrylate, or a softer type (100% butyl acrylate) is used with casein-based pigments. The first coat is applied to wet hog leathers after setting-out, before first drying. Improved adhesion of the film results. This method has been advocated also for other types of shoe upper leathers. New types of more lively pigments are needed. L.M.

Scudding of Glove Skins in the Drum. J. Turasek and V. Oplustil. *Kozarstvi*, **5**, 210–11 (1955).—Kidskin pelts are drummed with 50% H_2O and 0.5% of Synthapon L (sulfated lauryl alcohol) at 26°–28°C. for 1 hr. Sometimes "blind grain" results. Much more blind grain was found in skins degreased experimentally with 2% Synthapon L. Experiments proved that this product activates the enzymes of the bate. Samples of bated, unwashed pelt were treated with 100% water and 0, 0.5, 1, 1.5 and 2% of Synthapon L for 4 hr. at 37°C. Nitrogen was determined in the filtered liquors. The amounts found corresponded to 450, 500, 550, 900 and 1400 mg. collagen per 100 g. pelt. L.M.

Purification of Tannery Effluents. V. Kubelka and C. Halamek. *Kozarstvi*, **5**, 194–97, 215–17 (1955).—Wettings in Czechoslovakia amount to 40,000 tons of hides for vegetable, and 20,000 tons for chrome tanning annually. Tannery effluents (5 g. per l. dry solids) amount to about 1.6 million cubic meters. In a typical tannery, 36 l. of effluent were produced per kg. salted steer hide in vegetable tanning, and 72 l. in chrome tanning. Data are given on quantities of effluent from different operations, and the purification by sedimentation of concentrated and total effluent is discussed. L.M.

"Batex". L. Heller. *Kozarstvi*, **5**, 11–12 (1955).—Batex is a cement used for joining parts of shoe uppers and for other purposes in shoe manufacturing. Its composition is 76.7 parts chloroprene rubber latex (30% solids), 0.08 Latekol (a protective colloid), 6.0 water, 0.06 NH_4OH (25%), 8.0 colophonium, 1.16 hide glue, and 8.0 white spirit. Details of manufacture are described. L.M.

Enzymatic Bates. V. Pilc. *Kozarstvi*, **5**, 12–13 (1955).—A description of the production of enzymatic bates. Steer or hog pancreas, or the residue from the production of insulin, is disintegrated, mixed with sawdust, dried, and mixed with $(NH_4)_2SO_4$ (60–80% of the total weight of the bate). Products are made with 625, 833, abd 1250 Fuld-Gross units of enzyme activity by the Kubelkawagner method. The activity may decline by 20–40% after a year's storage. L.M.

The Relation Between the Raising of Domestic Animals and the Leather Industry. L. Masner. *Kozarstvi*, **5**, 54–7, 73–6 (1955).—World statistics of cattle raising and leather production are compared. The shoe industry, which consumes about 80% of the world's leather production, already uses 14% of leather substitutes to achieve a production that averages only one shoe per year for each of the earth's inhabitants. Further growth of shoe production is possible only through an increased production of leather substitutes. Relations between the live weights of various animals and the weights of their hides (7–8% for steer hides) are given. L.S.

Tan Yard to Tailor Shop. Anon. *Rohm & Haas Reporter*, **14**, No. 2, 2–7 (1956).—A description of an integrated, highly mechanized operation, whereby leather is tanned and made into garments in one factory. H.B.M.

Determination of Amino Acids by Ninhydrin Oxidation and Gas Chromatography. Separation of Leucine and Isoleucine. I. R. Hunter, K. P. Dimick, and J. W. Corse. *Chem and Ind.*, 1956, 294–5.—The volatile aldehydes formed by the action of ninhydrin on α-amino acids can be separated by gas chromatography. The separation of 3-methyl butanol produced from isoleucine and 2-methyl butanol produced from leucine has been demonstrated. Either of these aldehydes can be detected in a 1 : 10 mixture with the other. H.B.M.

National Hide Association Program. M. J. Parker. *Leather Mfr.* **73**, No. 4, 16–22 (1956).—An address. The Hide Association through its committee on small-packer hides has, after extensive study and work, evolved a proposed standard for hides from small packers. The Association is carrying out its grub-eradication program with increased intensity, attacking the program in the following manners: (1) use of a substance in feed or salt, (2) self-elimination of the grub through a peculiarity in its sex habits which causes the female to reproduce but once, and (3) injection of a solution which would enable cattle raisers to kill the grubs from within. Financial and active aid is being given by the Department of Agriculture on the grub-eradication problem. A voluntary test in the State of Washington over a 3-yr. period has shown elimination of 45% of the grubs. J.J.

Anionic Dyestuffs in Leather Coloring. J. A. Casnocha. *Leather Mfr.*, **73**, No. 4 24–38 (1956).—An outline of the anionic dyes of the azo, triarylmethane, heteropolycyclic and anthraquinone classes, including a brief history of the various classes, methods of preparing the dyes in the group, their structure formulas, and color. In some cases the properties of the dyes are given with respect to fastness, crocking, penetration, and how various salts affect the color. Also discussed are the good and bad features of the dye or group, the pH of 0.2% solution of dyes, and how temperature affects some dyes. J.J.

Proposed Small-Packer Standards. National Hide Association. *Leather Mfr.*, **73,** No. 4, 60–62 (1956).—The proposed small-packer standards give definitions of what constitutes No. 1, No. 2, and No. 3 hides, selection of hides, manure allowance, grub allowance, proper cure, tare allowance, and range for hide weights on average weight of hide purchased.

J.J.

PATENT

Condensation Product. DEHYDAG Deutsche Hydrierwerke G.m.b.h. (Erik Schirm, inventor). Ger. Pat. 859,460. Dec. 15, 1952 (cl.12$_o$, 23$_{02}$); through *Chem. Abstr.*, **50,** 1907*i*.—Sulfite waste liquor condensed with aromatic sulfonamides in the presence of aldehydes or substances yielding aldehydes, or with *N*-(hydroxymethyl) compounds of sulfonamides, or their H halide esters, give condensation products useful as tanning agents. The waste sulfite liquor, freed from Ca and Fe compounds, is evaporated to dryness, 640 parts of the dried waste sulfite liquor is slurried with 360 parts by weight of water, and the syrupy mass is treated with 250 parts of *p*-$(CH_3)C_6H_4SO_2NHCH_2OH$ at 90°C. until a sample dissolves clear in water. For tanning purposes the condensation product is diluted with water and adjusted to a suitable pH value. The products obtained by condensing waste sulfite liquor with 1,3- and 1,4-$C_6H_4(SO_2NH_2)_2$ and HCHO, or with 1,3,5- or 1,3,6-$C_{10}H_5(SO_2NH_2)_3$ and HCHO are likewise suitable substitutes for tanning agents. A second patent by the same inventor (Ger. 877,758, May 25, 1953 [*Chem. Abstr.*, **50,** 4539*f*]) describes the carrying out of the condensation at pH 7 to 8, whereby an increased yield of tanning material is obtained. A third patent (Ger. 877,759) covers the replacement of sulfite waste liquor or lignosulfonic acids in the above condensation by similar compounds containing solublizing radicals and in which compound alkylol groups can be introduced, such as phenols, ketones, aldehydes, carboxamides, sulfonamides or urea derivatives, substituted by SO_3H or COOH radicals.

NEW PUBLICATIONS

Industrial Research Laboratories of the United States. 10th ed., 1956. Compiled and published by the National Academy of Sciences, National Research Council, Publications Office, 2101 Constitution Ave., Washington, D. C. Contains data on 4,834 laboratories of 4,060 companies, with an index of research activities including over 1000 major subject headings. 560 pp. $10.

Leather Finishes. J. S. Mudd. 2nd ed., 1955 Croydon, England: A. Harvey. 15 shillings.

Production of Leathers Resistant to Powerful Oxidants and Reductants. PB 111882. Washington, D. C.: Office of Technical Services, U. S. Department of Commerce. 62 pp. $1.75.

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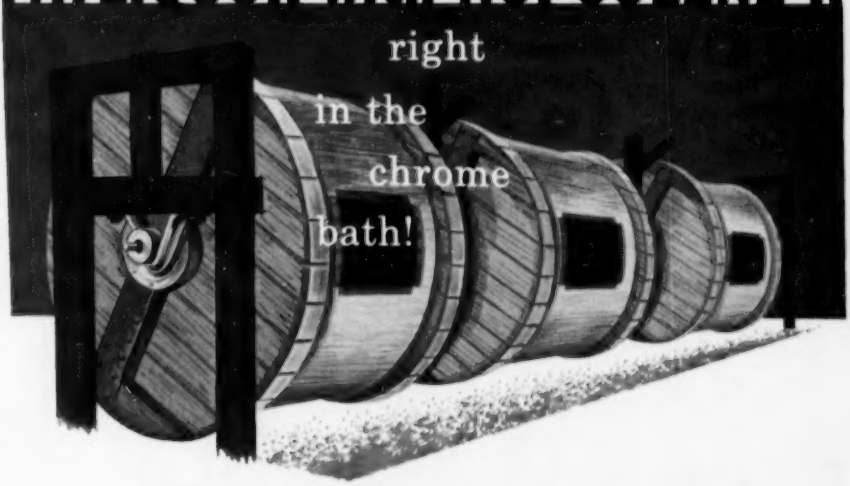

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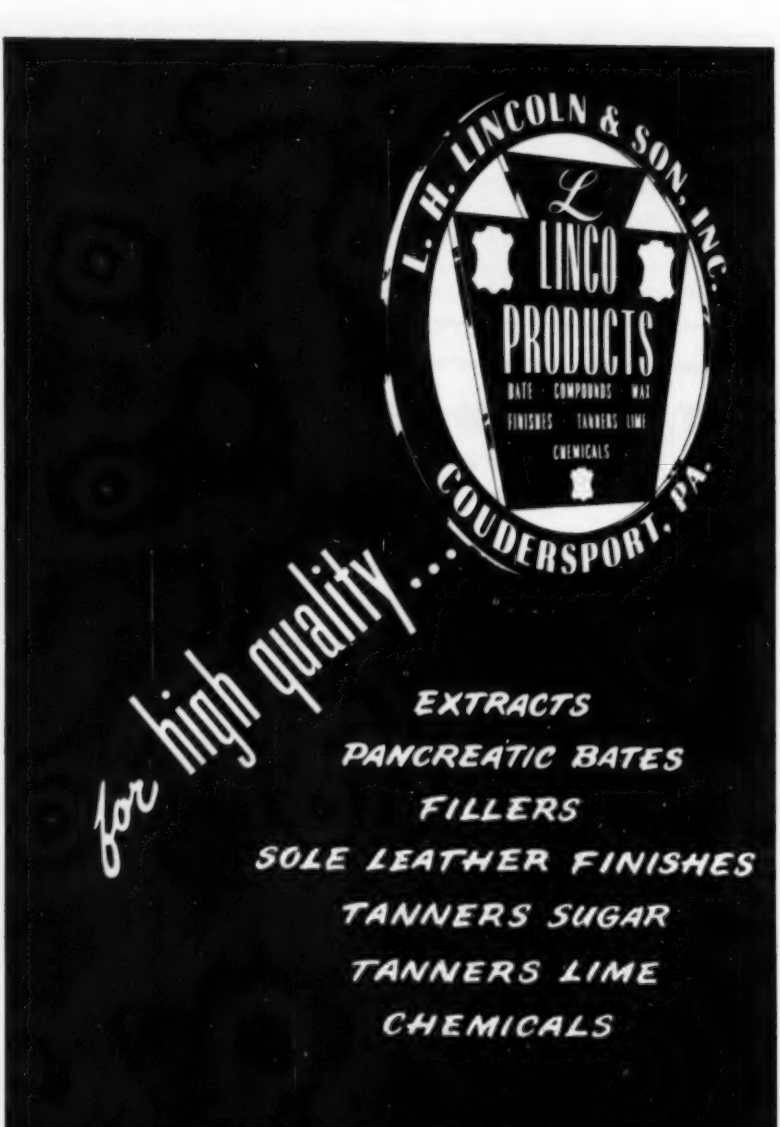
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INDEX TO ADVERTISERS

ADOLF SCHUBERT XXXIII
AMERICAN CYANAMID COMPANY, Dyestuff Department XVII
AMERICAN EXTRACT CO. ..... Outside Back Cover
APEX CHEMICAL CO., INC. XXXII
ARKANSAS COMPANY, INC. VII
ARTHUR C. TRASK CO. XXIX
ARTHUR THOMAS CO. XX
ATLAS REFINERY XVI
BARIUM REDUCTION CORPORATION XXIII
BARKEY IMPORTING CO., INC. XIV
BONA ALLEN, INC. XXXIII
BUCKMAN LABORATORIES, INC. XII
CHEMTAN COMPANY XIX
CLINTON CORN PROCESSING COMPANY XXXIV
EISENDRATH TANNING CO. XXV
FRANK CRYSTAL III
FRANK F. MARSHALL XXIX
GARDEN STATE TANNING INC. XXIX
GEIGY COMPANY, INC. IX
HOOKER ELECTROCHEMICAL CO. XI
HOWES LEATHER CO., INC. XXXIII
INTERNATIONAL PRODUCTS CORP. X
KEPEC CHEMICAL CORPORATION XXIX
LEATHER AND SHOES VI
LEATHER MANUFACTURER, THE XXVII
L. H. LINCOLN & SON, INC. XXX
MARDEN WILD CORPORATION XXIV
MUTUAL CHEMICAL DIVISION, ALLIED CHEMICAL & DYE CORP. ..... Inside Back Cover
NEWARK LEATHER FINISH CO. XXXI
NOPCO CHEMICAL CO. XXI
OHIO LEATHER CO., THE XXV
OLSON IMPORTING COMPANY, INC., THE ..... Inside Front Cover
PACIFIC COAST BORAX CO. XXVI
PRESTO COLOR CO. XXVI
REILLY-WHITEMAN-WALTON CO. XXVII
RIVER PLATE CORPORATION, THE IV
ROBESON PROCESS CO. II
ROHM & HAAS I
SALEM OIL & GREASE CO. XXVIII
SAXE CUTCH CORPORATION XXIV
SHOE & LEATHER REPORTER XXVIII
SOLVAY PROCESS DIVISION, ALLIED CHEMICAL & DYE CORP. V
SOUTH AFRICAN WATTLE EXTRACT MFRS. ASSO. XV
SUN OIL COMPANY XXIV
TANEXCO, INC. XXXVI
TANIMEX CORPORATION XIII
TANNER'S COUNCIL RESEARCH LABORATORY ..... XXIX—XXXIII—XXXIV
UNION STARCH & REFINING CO. XXXII
UNITED STATES TESTING COMPANY XXXI
WALLERSTEIN COMPANY, INC. XXII
WARNER CO. XXV
WHITTEMORE-WRIGHT CO. XXVI
WOLF, JACQUES & CO. VIII
YOUNG CO., J. S. XVIII

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